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Triphenylarsenic(V) and -antimony(V) derivatives of multidentate Schiff bases: synthesis, characterization, and antimicrobial activities

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Equimolar reactions of $\text{Ph}_n\text{M}(\text{OPr}^i)_2$ (where $\text{M} = \text{As}$ and Sb) with Schiff bases $[\text{OHC}_6\text{H}_4\text{CH}=\text{N}(\text{R})\text{OH}]$ in benzene solution yield organoarsenic and -antimony derivatives, $\text{Ph}_n\text{M}\{\text{OC}_6\text{H}_4\text{CH}=\text{N}(\text{R})\text{O}\}_n$ (where $\text{M} = \text{As}$ and Sb ; $n = 1$ and 3 ; $\text{R} = -\text{CH}_2\text{CH}(\text{CH}_3)-$, $-(\text{CH}_2)_3-$, $-(\text{CH}_2)_2-$, and $-\text{C}(\text{CH}_3)_2\text{CH}_2-$). All these derivatives have been characterized by elemental analyses and molecular weight measurements, and structures have been proposed on the basis of IR, NMR (^1H and ^{13}C), and FAB-mass studies. Schiff bases and their corresponding organoantimony derivatives have been screened for antimicrobial activity against *Aspergillus flavus* (fungus) and *Escherichia coli* (bacteria).

Keywords: Triphenylarsenic(V); Triphenylantimony(V); Bifunctional tridentate Schiff bases; FAB-mass; Antimicrobial activities

1. Introduction

Organoarsenic and -antimony +5 oxidation state have applications in the medicinal field [1, 2], in catalysis [3, 4], as precursors for superconducting materials in MOCVD process [5] and as biocides [6]. These compounds [7] may exhibit different behavior than their corresponding +3 oxidation state [8] due to enhanced coordination ability.

Tridentate Schiff-base ligands derived from amino alcohols are reported to show antibacterial and antifungal activities [9, 10]. Some organoantimony derivatives with multidentate Schiff bases in +3 and +5 oxidation states have antimicrobial activities [11, 12]. Therefore, it was thought worthwhile to bring these two biologically active moieties together in a molecule. Therefore, in this article, we report the synthesis and

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characterization of triphenylarsenic(V) and -antimony(V) derivatives of bifunctional tridentate Schiff bases.

2. Experimental

Solvents were dried by standard methods [13] before use. Ph_3MBr_2 [14] (where $\text{M} = \text{As}, \text{Sb}$), PhAsCl_2 [15], PhSbCl_2 [16], and Schiff bases [17] have been prepared by literature methods.

All these organoarsenic and -antimony derivatives have been synthesized by a similar method; for convenience, the synthesis of one representative compound of the series is given in detail. The synthetic and analytical data of analogous derivatives have been summarized in table 1.

Table 1. Synthetic, analytical, and physical data of 1-4 and 6-9 derivatives.

Compound	Reactants g (mmol)	Empirical formula (Yield %) physical state (m.p. °C)			Molecular weight found (Calcd)	Analyses % found (Calcd)	
		Na	Ph_3MBr_2	LH_2		M	N
1	0.14 (3.04)	M=As 1.45 (3.11)	L^1H_2 0.57 (3.18)	$\text{C}_{28}\text{H}_{26}\text{AsNO}_2$ (92) Light brown viscous liquid	480.01 (483.43)	M=As 15.52 (15.49)	2.86 (2.89)
2	0.13 (2.83)	M=As 1.35 (2.90)	L^2H_2 0.52 (2.90)	$\text{C}_{28}\text{H}_{26}\text{AsNO}_2$ (94) Light brown viscous liquid	496.33 (483.43)	M=As 15.48 (15.49)	2.85 (2.89)
3	0.14 (3.05)	M=As 1.46 (3.13)	L^3H_2 0.52 (3.15)	$\text{C}_{27}\text{H}_{24}\text{AsNO}_2$ (95) Brown viscous liquid	456.07 (469.41)	M=As 15.93 (15.96)	2.95 (2.98)
4	0.14 (3.05)	M=As 1.40 (3.00)	L^4H_2 0.58 (3.00)	$\text{C}_{29}\text{H}_{28}\text{AsNO}_2$ (94) Brown viscous liquid	483.93 (497.46)	M=As 15.01 (15.06)	2.83 (2.81)
6	0.13 (2.83)	M=Sb 1.46 (2.85)	L^1H_2 0.51 (2.84)	$\text{C}_{28}\text{H}_{26}\text{SbNO}_2$ (93) Yellow solid (140)	524.41 (530.25)	M=Sb 22.86 (22.96)	2.60 (2.64)
7	0.12 (2.61)	M=Sb 1.34 (2.61)	L^2H_2 0.47 (2.62)	$\text{C}_{28}\text{H}_{26}\text{SbNO}_2$ (92) Yellow solid (58)	528.16 (530.25)	M=Sb 22.92 (22.96)	2.65 (2.64)
8	0.13 (2.83)	M=Sb 1.49 (2.90)	L^3H_2 0.48 (2.91)	$\text{C}_{27}\text{H}_{24}\text{SbNO}_2$ (94) Yellow solid (60)	505.02 (516.22)	M=Sb 23.52 (23.58)	2.68 (2.71)
9	0.13 (2.83)	M=Sb 1.41 (2.75)	L^4H_2 0.53 (2.74)	$\text{C}_{29}\text{H}_{28}\text{SbNO}_2$ (93) Yellow solid (63)	559.21 (544.28)	M=Sb 22.39 (22.37)	2.58 (2.57)

$\text{L}^1\text{H}_2 = \text{HOC}_6\text{H}_4\text{CH}=\text{NCH}_2\text{CH}(\text{CH}_3)\text{OH}$; $\text{L}^2\text{H}_2 = \text{HOC}_6\text{H}_4\text{CH}=\text{N}(\text{CH}_2)_3\text{OH}$; $\text{L}^3\text{H}_2 = \text{HOC}_6\text{H}_4\text{CH}=\text{N}(\text{CH}_2)_2\text{OH}$ and $\text{L}^4\text{H}_2 = \text{HOC}_6\text{H}_4\text{CH}=\text{NC}(\text{CH}_3)_2\text{CH}_2\text{OH}$.

Compound No.	Carbon Found (Calcd)	Hydrogen Found (Calcd)
6	63.46 (63.37)	4.96 (4.94)
8	62.59 (62.77)	4.70 (4.68)

2.1. Synthesis of triphenylarsenic(V) derivatives [$\text{Ph}_3\text{As}(\text{OC}_6\text{H}_4\text{CH}=\text{NCH}_2\text{CH}(\text{CH}_3)\text{O})$]

A weighed amount of sodium metal (0.14 g, 3.04 mmol) was added to ~20 mL dried isopropanol and the mixture was stirred for ~1 h and benzene solution of Ph_3AsBr_2 (1.45 g, 3.11 mmol) was added. The reaction mixture was refluxed for 4 h; NaBr thus precipitated was filtered off. A benzene solution of $\{\text{OHC}_6\text{H}_4\text{CH}=\text{NCH}_2\text{CH}(\text{CH}_3)\text{OH}\}$ (0.57 g, 3.18 mmol) was mixed to the filtrate ($\text{Ph}_3\text{Sb}(\text{OPr}^i)_2$) and the reaction mixture was refluxed for ~5 h under a fractionating column. On completion of the reaction when the azeotrope showed negligible amount of isopropanol, the reaction was stopped and excess solvent was removed under reduced pressure to get a viscous brown product. For purification, this viscous compound was dissolved in a small amount of benzene and then hexane was added till the compound began to separate. After decanting off the solvent, the compound was dried under vacuum (yield 92%). Anal. Calcd for $\text{C}_{28}\text{H}_{26}\text{AsNO}_2$ (%): As, 15.49; N, 2.89. Found: As, 15.52; N, 2.86.

For $\text{PhAs}(\text{OC}_6\text{H}_4\text{CH}=\text{NCH}_2\text{CH}(\text{CH}_3)\text{O})$ (**5**), Na (0.21 g, 4.56 mmol), PhAsCl_2 (1.02 g, 4.57 mmol), and $\text{OHC}_6\text{H}_4\text{CH}=\text{NCH}_2\text{CH}(\text{CH}_3)\text{OH}$ (0.82 g, 4.5 mmol). Anal. Calcd for $\text{C}_{16}\text{H}_{16}\text{AsNO}_2$ (dark yellow viscous liquid) (%): As, 22.75; N, 4.25. Found: As, 21.98; N, 4.22.

For $\text{PhSb}(\text{OC}_6\text{H}_4\text{CH}=\text{NCH}_2\text{CH}(\text{CH}_3)\text{O})$ (**10**), Na (0.18 g, 3.98 mmol), PhSbCl_2 (1.07 g, 3.98 mmol), and $\text{OHC}_6\text{H}_4\text{CH}=\text{NCH}_2\text{CH}(\text{CH}_3)\text{OH}$ (0.71 g, 3.99 mmol). Anal. Calcd for $\text{C}_{16}\text{H}_{16}\text{SbNO}_2$ (yellow viscous liquid) (%): Sb, 32.38; N, 3.72. Found: Sb, 32.40; N, 3.75.

2.2. Physical measurements

Both antimony and arsenic have been analyzed by the iodometric method [18] and nitrogen has been estimated by the Kjeldhal method [18]. Isopropanol in the azeotrope has been determined oxidimetrically [19]. Molecular weights have been determined cryoscopically in freezing benzene solution using a Beckmann's thermometer. Infrared (IR) spectra of these compounds have been recorded as liquid film or Nujol mull using KBr cells from 4000 to 400 cm^{-1} on an FTIR spectrophotometer model 8400 S Shimadzu. ^1H - and ^{13}C -NMR spectra have been recorded in CDCl_3 solution on a JEOL-FT AL 300 MHz spectrometer using TMS as internal reference. FAB-mass spectra of **2** and **8** were recorded on a MICROMASS QUATRO II triple quadrupole mass spectrometer.

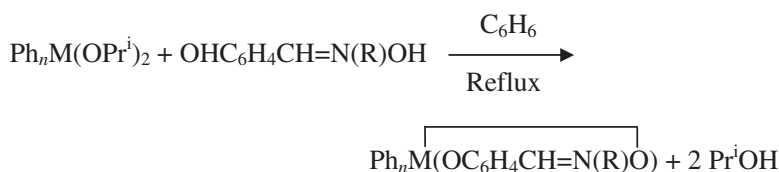
2.3. Antimicrobial activities

The agar diffusion method was used to observe the antimicrobial activities of free Schiff base and their corresponding organoantimony derivatives. In this method, sterilized nutrient agar medium and Whatman no. 1 paper disc (6 mm in diameter) were used; 0.1 mL inoculums of the test organism were spread uniformly on the surface of PDA (Potato, Dextrose Agar for Fungus) and NA (Nutrient Agar for Bacteria) medium in a petriplate using a spreader. Test samples were prepared in 1% and 5% concentration by adding 0.01 g and 0.05 g compounds, respectively, in 1 mL of methanol. Paper discs were dipped into the solution and then placed on the surface of the agar plates.

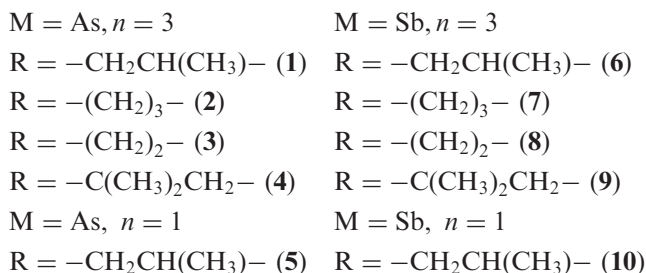
The plates were incubated at 28°C for 24 h. During incubation, the compounds diffused from the filter paper into the medium. The activities of ligands and their corresponding organoantimony derivatives were assessed by measuring the diameter of inhibition zone. The results were compared against those of control, which was screened simultaneously. Table 4 presents the inhibition zone in diameter. The readings are the mean value of three replicates.

3. Results and discussion

Reactions of $\text{Ph}_n\text{M}(\text{OPr}^i)_2$ ($\text{M} = \text{As}$ and Sb) (prepared *in situ* by reactions of Ph_3MX_2 ($\text{X} = \text{Br}$ or Cl) and NaOPr^i in 1 : 2 molar ratio) with Schiff bases in equimolar ratio in refluxing benzene give corresponding organoarsenic and -antimony derivatives.



where



All these compounds are colored viscous liquids or solids and are soluble in common organic solvents. All these derivatives are monomeric in freezing benzene solution. Compounds **1–10** have been characterized by IR, ^1H - and ^{13}C -NMR, and FAB-mass studies.

3.1. IR spectra

A comparative study of IR spectra of these compounds with their corresponding Schiff bases shows disappearance of absorption bands for alcohol $-\text{OH}$ as well as phenol $-\text{OH}$, observed as a broad band at $3200\text{--}3630\text{ cm}^{-1}$ in the spectra of free ligands, indicating that both phenol and alcohol are deprotonated during chelation. A new band at $450\text{--}475\text{ cm}^{-1}$ (in spectra of **1–5**) and $530\text{--}580\text{ cm}^{-1}$ (in spectra of **6–10**) may be assigned to $\nu(\text{As}-\text{O})$ [20] and $\nu(\text{Sb}-\text{O})$ [21], respectively.

Bands at $1520\text{--}1610\text{ cm}^{-1}$, $1021\text{--}1070\text{ cm}^{-1}$, and $1230\text{--}1300\text{ cm}^{-1}$ in **1–10** have been assigned to $\nu(\text{C}=\text{N})$ [10], alcohol $\nu(\text{C}-\text{O})$ [22], and phenol $\nu(\text{C}-\text{O})$ [22], respectively, with a shift of $35\text{--}45\text{ cm}^{-1}$ to lower wavenumber compared to their position in spectra of free ligands. The shift in position of these bands indicates involvement of all three groups in

bonding, thereby suggesting C=N, alcohol >C–O, and phenol >C–O in tridentate chelation. New bands at 420–445 cm⁻¹ and 420–435 cm⁻¹ have been assigned to $\nu(\text{As}\leftarrow\text{N})$ [23] and $\nu(\text{Sb}\leftarrow\text{N})$ [24], respectively. Bands due to As–C [25] and Sb–C [26] have been observed at 440–460 cm⁻¹ and 465–505 cm⁻¹, respectively, in the spectra of these derivatives.

3.2. ¹H-NMR spectra

The characteristic signals in the ¹H-NMR spectra of all these derivatives are summarized in table 2. Signals at δ 3.80–4.39 ppm and δ 9.82–10.67 ppm due to alcohol –OH and phenol –OH, respectively, in the free ligands are absent in **1–10**. This indicates deprotonation of ligand during formation of these derivatives. The imine proton –CH=N signal is observed at δ 8.21–8.38 ppm in spectra of **1–5** and at δ 8.02–8.40 ppm in spectra of **6–10**, showing a small shift in **1–10** compared to its position in spectra of Schiff bases. This indicates that nitrogen imine is involved in the coordination with metal.

Table 2. ¹H-NMR spectral data of organoarsenic and -antimony derivatives, **1–10** (in δ ppm).

Compound	R (<i>J</i> in Hz)	CH=N	(C ₆ H ₅) ₃ -M	Phenyl group (OC ₆ H ₄ CH=N)
1	1.17d, 3H(CH ₃); (<i>J</i> = 12.0) 3.55, d, 2H(CH ₂); (<i>J</i> = 18.0) 3.96–4.15, m, H(CH)	8.29	7.50–7.96, m, 15H	6.81–7.48, m, 4H
2	1.15–1.17, m, 2H(CH ₂) 1.85–1.89, t, 2H(NCH ₂); (<i>J</i> = 6.31) 3.61–3.66, t, 2H(OCH ₂); (<i>J</i> = 6.01)	8.21	7.51–7.71, m, 15H	6.78–7.49, m, 4H
3	3.72–3.76, t, 2H(CH ₂); (<i>J</i> = 6.00) 3.88–3.91, t, 2H(OCH ₂); (<i>J</i> = 6.00)	8.38	7.55–7.72, m, 15H	6.86–7.54, m, 4H
4	1.16, s, 6H(CH ₃) 2.47, s, 2H(CH ₂)	8.36	7.48–7.84, m, 15H	6.78–7.43, m, 4H
5	1.10, d, 3H(CH ₃); (<i>J</i> = 6.30) 2.69, d, 2H(CH ₂); (<i>J</i> = 9.01) 3.64–3.89, m, H(CH) 1.36, d, 3H(CH ₃); (<i>J</i> = 6.01)	8.24	7.42–7.69, m, 15H	6.73–7.22, m, 4H
6	3.49, d, 2H(CH ₂); (<i>J</i> = 9.01) 4.01–4.18, m, H(CH)	8.40	7.65–7.88, m, 15H	6.96–7.36, m, 4H
7	1.97–2.07, m, 2H(CH ₂) 3.56–3.59, t, 2H(NCH ₂); (<i>J</i> = 6.00) 4.09–4.12, t, 2H(OCH ₂); (<i>J</i> = 3.01)	8.18	7.47–7.82, m, 15H	6.54–7.25, m, 4H
8	3.65–3.68, t, 2H(NCH ₂); (<i>J</i> = 6.00) 4.12–4.16, t, 2H(OCH ₂); (<i>J</i> = 6.00)	8.35	7.47–7.90, m, 15H	6.71–7.35, m, 4H
9	1.11, s, 6H(CH ₃) 3.77, s, 2H(CH ₂)	8.02	7.52–7.80, m, 15H	7.15–7.40, m, 4H
10	1.22, d, (CH ₃); (<i>J</i> = 6.00) 3.53, d, 2H(CH ₂); (<i>J</i> = 12.01) 3.91–4.12, m, H(CH)	8.21	7.59–7.88, m, 15H	6.94–7.34, m, 4H

d, doublet; t, triplet; m, multiplet.

The phenyl ring protons attached to As or Sb are a multiplet at δ 7.48–7.96 ppm in all these derivatives.

3.3. ^{13}C -NMR spectra

^{13}C -NMR chemical shifts of these derivatives are summarized in table 3. ^{13}C -NMR spectra exhibit carbon signals for phenol $>\text{C}-\text{O}$ and alcohol $>\text{C}-\text{O}$ at δ 165.0–168.9 ppm and δ 63.5–70.3 ppm in **1–5** and δ 160.9–166.2 ppm and δ 61.9–71.5 ppm, respectively, in **6–10**. These carbon signals show small downfield shifts (\sim 4–5 ppm) compared to free ligands. This indicates involvement of these groups in bonding. Signal for imine carbon ($\text{CH}=\text{N}$) at δ 161.1–165.6 ppm in **1–5** and δ 148.2–154.5 ppm in **6–10** shows a small shift in comparison to free ligand, indicating involvement in bonding. Signals for phenyl carbon attached to central metal are at δ 128.4–139.5 ppm and δ 129.5–148.2 ppm in **1–5** and **6–10**, respectively.

The corrected chemical shift value [27] δ' and Hammett–Taft's constant [28], σR° , for phenyl carbons attached to As or Sb in **1–10** have been calculated using the relation $\delta' = \text{C}_p - \text{C}_m$ (where δ C_p and δ C_m are the chemical shifts of para and meta carbons of phenyl attached to metal) and the equation $\delta' = 23.06 \sigma R^\circ$, respectively.

The δ' and σR° values of **1–5** and **6–10** are found negative in the range δ –3.36 to –1.18 ppm and δ –0.15 to –0.05 ppm and δ –1.91 to –0.73 ppm and δ –0.03 to –0.08 ppm, respectively. More negative values of δ' and σR° in arsenic derivatives reveal that electron release through $d\pi$ – $p\pi$ conjugation is more in arsenic derivatives, perhaps due to the smaller size of As which makes it more suitable for $d\pi$ – $p\pi$ conjugation. These observations conform with observed metal–carbon bond energies which are in the order of As–C $>$ Sb–C [29].

3.4. FAB-mass spectra

Mass spectra of **2** and **8** reveal the monomeric nature of these derivatives. The fragmentation patterns of these compounds are summarized in “Supplementary material.”

On the basis of above-mentioned physico-chemical and IR, ^1H , ^{13}C , and FAB-mass spectral evidences, it is clear that the ligands are bifunctional tridentate in all these derivatives. In view of the monomeric nature of these derivatives and the presence of three phenyl rings on central arsenic or antimony atom, a structure (figure 1) in which central atom acquires octahedral geometry [30, 31] may be tentatively proposed for all these derivatives.

3.5. Antimicrobial activity

LH_2 and organoantimony derivatives, **6–10**, have been screened for antimicrobial activity against *Aspergillus flavus* (fungus) and *Escherichia coli* (bacteria). *Mycostatin* and *gentamicin* have been used as standard antibiotics and results have been compared with those of ligands and their organoantimony derivatives. The results (table 4)

Table 3. ^{13}C -NMR spectral data of triphenylarsenic(V) and -antimony(V) derivatives, **1–8** (in δ ppm).

Compound	Alkylene group	Imine carbon (CH=N)	Phenolic carbon (C–O)	Phenyl carbon (OC ₆ H ₄ CH=N)	(C ₆ H ₅) ₃ -M	δ'	σR°
1	27.2 (CH ₃)	162.1	166.1	117.0–132.3	139.5(i) 133.6(o) 132.5(m) 130.1(p)	–2.38	–0.10
	63.8 (CH ₂ N)						
	66.9 (OCH)						
2	25.3 (CH ₂)	161.5	165.0	116.8–132.3	139.5(i) 133.6(o) 131.9(m) 128.6(p)	–3.36	–0.15
	55.8 (CH ₂ N)						
	63.5 (OCH ₂)						
3	60.8 (CH ₂ N)	165.6	166.2	116.9–133.6	139.5(i) 136.6(o) 129.6(m) 128.4(p)	–1.18	–0.05
	63.8 (OCH ₂)						
4	25.3 (CH ₃)	161.9	168.9	111.6–132.6	136.6(i) 133.6(o) 130.4(m) 128.6(p)	–1.80	–0.08
	60.8 {C(CH ₃)}						
	70.3 (OCH ₂)						
5	25.2 (CH ₃)	161.5	165.2	116.98–131.23	132.2(i) 130.7(o) 130.4(m) 128.8(p)	–1.59	–0.07
	55.7 (CH ₂ N)						
	64.2 (OCH)						
6	21.6 (CH ₃)	153.8	166.1	117.3–135.5	147.2(i) 133.5(o) 132.9(m) 131.0(p)	–1.91	–0.08
	60.8 (CH ₂ N)						
	65.7 (OCH)						
7	32.9 (CH ₂)	150.0	166.2	117.0–134.6	147.2(i) 135.2(o) 133.4(m) 132.1(p)	–1.31	–0.06
	59.9 (CH ₂ N)						
	64.4 (OCH ₂)						
8	54.5 (CH ₂ N)	153.1	165.8	117.0–135.2	135.5(i) 134.8(o) 133.1(m) 132.4(p)	–0.73	–0.03
	61.9 (OCH ₂)						
9	27.5 (CH ₃)	154.5	164.0	117.5–135.3	135.6(i) 133.8(o) 131.1(m) 129.5(p)	–1.65	–0.07
	60.4 {C(CH ₃)}						
	71.5 (OCH ₂)						
10	20.3 (CH ₃)	148.2	160.9	116.2–134.6	148.2(i) 132.9(o) 131.5(m) 129.9(p)	–1.57	–0.06
	61.7 (CH ₂ N)						
	64.1 (OCH)						

indicate that the metal chelates have higher activity than the free ligands and their activity increases as concentration increases. The antimony derivatives have antimicrobial activity but less than that of standard antibiotics. This increased activity of the metal chelate as compared to the free ligand moieties can be explained as Tweedy's chelation theory [32] and the concentration [33].

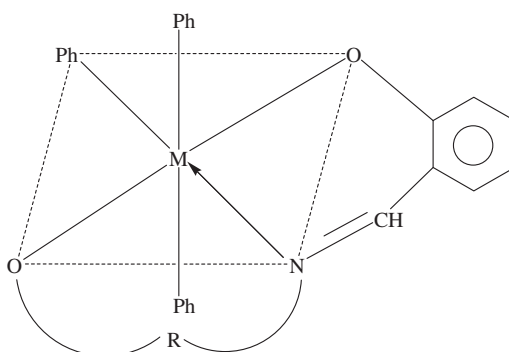


Figure 1. Proposed structure of the triorganoarsenic(V) and -antimony(V) derivatives (M = As and Sb).

Comparison of the antimicrobial activities of the Schiff bases and triphenylantimony(V) derivatives with known antibiotics and phenyl antimony(III) derivatives shows that (1) **6** exhibits greater antibacterial effect toward *E. coli* than **1** and **10**, but less than gentamicin and (2) **6** exhibits greater antifungal effect toward *A. flavus* than **1** and **10**, but less than mycostatin.

The increased activity of antimony compounds in +5 oxidation state may be explained from the presence of three phenyl groups [12] which generally damage the cell wall of bacteria and fungi by reacting with peptides, but the exact mechanism is yet to be established.

Complexation of antimony derivatives with biologically active Schiff bases results in increased activity. The antimicrobial activities of the free ligands and triphenylantimony(V) derivatives against fungus (*A. flavus*) and bacteria (*E. coli*) are shown in "Supplementary material."

4. Conclusions

From spectroscopic evidence, the ligand is bifunctional tridentate leading to the structure in figure 1. To compare these arsenic(V) and antimony(V) compounds with compounds in +3 oxidation state, we have synthesized two representative compounds of arsenic and antimony in +3 oxidation state; the coordination pattern of ligand in both cases (+3 and +5) remains the same. Strong electron attracting tendency of arsenic and antimony in +5 oxidation state is reflected by more downfield shift of C=N and >C=O and alkylene group carbons in ^{13}C -NMR spectra with a similar trend observed in ^1H -NMR spectra. The σR° and δ' value for As(V) and Sb(V) compounds are more negative than in arsenic(III) and Sb(III) compounds, which indicates more electron transfer from central metal to phenyl.

Biological activities of the Schiff bases and their corresponding organoantimony derivatives have been studied by the agar diffusion method [34] on various microorganisms. Organoantimony derivatives are more active than free ligands. Triphenylantimony(V) derivatives show higher antimicrobial activity compared to phenylantimony(III) derivatives.

Table 4. Antimicrobial studies of ligands and their triphenylantimony(V) derivatives showing inhibition zone after 2–3 days (in diameter).

Compound No.	Ligands/antimony derivatives	Inhibition zone (cm)				
		Concentration (%)	Standard antibiotic			
			<i>Mycostatin</i> (fungus)	<i>Gentamicin</i> (bacteria)	<i>A. flavus</i> (fungus)	<i>E. coli</i> (bacteria)
L ¹ H ₂	HOC ₆ H ₄ CH=NCH ₂ CH(CH ₃)OH	1	1.4	1.2	0.83 ± 0.07	0.71 ± 0.03
L ² H ₂	HOC ₆ H ₄ CH=N(CH ₂) ₃ OH	5	2.5	2.0	1.10 ± 0.06	1.00 ± 0.08
L ³ H ₂	HOC ₆ H ₄ CH=N(CH ₂) ₂ OH	1	1.4	1.2	0.73 ± 0.03	0.90 ± 0.03
L ⁴ H ₂	HOC ₆ H ₄ CH=N(CH ₃) ₂ CH ₂ OH	5	2.5	2.0	0.91 ± 0.03	1.20 ± 0.06
		1	1.4	1.2	0.00 ± 0.00	0.74 ± 0.03
		5	2.5	2.0	0.84 ± 0.03	0.82 ± 0.01
		1	1.4	1.2	0.70 ± 0.03	1.10 ± 0.06
		5	2.5	2.0	0.92 ± 0.00	1.20 ± 0.01
6	Ph ₃ Sb[OC ₆ H ₄ CH=NCH ₂ CH(CH ₃)O]	1	1.4	1.2	0.80 ± 0.05	1.10 ± 0.06
7	Ph ₃ Sb[OC ₆ H ₄ CH=N(CH ₂) ₃ O]	5	2.5	2.0	1.32 ± 0.08	1.40 ± 0.06
		1	1.4	1.2	0.80 ± 0.05	1.00 ± 0.06
		5	2.5	2.0	1.10 ± 0.05	1.30 ± 0.06
8	Ph ₃ Sb[OC ₆ H ₄ CH=N(CH ₂) ₂ O]	1	1.4	1.2	0.60 ± 0.05	0.80 ± 0.06
		5	2.5	2.0	1.30 ± 0.05	1.11 ± 0.06
9	Ph ₃ Sb[OC ₆ H ₄ CH=NC(CH ₃) ₂ CH ₂ O]	1	1.4	1.2	1.00 ± 0.05	1.20 ± 0.01
		5	2.5	2.0	1.20 ± 0.05	1.50 ± 0.06
10	Ph ₃ Sb[OC ₆ H ₄ CH=NCH ₂ CH(CH ₃)O]	1	1.4	1.2	0.93 ± 0.05	1.02 ± 0.06
		5	2.5	2.0	1.20 ± 0.08	1.25 ± 0.06

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References

- [1] N.N. Greenwood. *Chemistry of Elements*, p. 694, Pergamon Press, London (1984).
- [2] C. Silvestru, I. Haiduc, E.R.T. Tiekink, D. deVos, M. Biesemans, R. Willem, M. Gielen. *Appl. Organomet. Chem.*, **9**, 597 (1995).
- [3] Q.L. He, E.B. Wang. *Inorg. Chim. Acta*, **298**, 235 (2000).
- [4] D.L. Mckey. *Chem. Abstr.*, **87**, 157206 (1977).
- [5] K.T. Higa, C. George. *Organometallics*, **9**, 275 (1990).
- [6] C. Silvestru, M. Curtui, I. Haiduc, M.J. Begley, D.B. Sowerby. *J. Organomet. Chem.*, **426**, 49 (1992).
- [7] P.K. Sharma, A.K. Rai, Y.P. Singh. *Heteroatom Chem.*, **18**, 76 (2007).
- [8] S. Sharma, R.K. Sharma, A.K. Rai, Y.P. Singh. *Heteroatom Chem.*, **15**, 92 (2004).
- [9] V. Vajpayee, Y.P. Singh, D. Nandani, A. Batra. *Appl. Organomet. Chem.*, **21**, 694 (2007).
- [10] C. Jayabalakrishanan, K. Natarajan. *Transition Met. Chem.*, **27**, 75 (2002).
- [11] M.K. Khosa, M. Mazhar, S. Ali, K. Shahid, F. Malik. *Turk. J. Chem.*, **30**, 345 (2006).
- [12] R. Kant, G. Ameesh, K. Chandrashekar, S.K. Shukla. *Phosphorus, Sulfur Silicon Relat. Elem.*, **184**, 2453 (2009).
- [13] D.D. Perrin, W.L.F. Armearago, D.R. Perrin. *Purification of Laboratory Chemicals*, 2nd Edn, Pergamon Press, New York (1980).
- [14] A.D. Beveridge, G.S. Harris, F. Inglis. *J. Chem. Soc. A*, 520 (1966).
- [15] R.L. Barker, E. Booth, W.E. Jones, A.F. Mollidge, F.N. Woodward. *J. Chem. Soc. Ind. (London)*, **68**, 285 (1949).
- [16] M. Nunn, D.B. Sowerby, D.M. Wesolek. *J. Organomet. Chem.*, **251**, C45 (1983).
- [17] H.B. Singh, J.P. Tandon. *J. Indian Chem. Soc.*, **LVIII**, 836 (1981).
- [18] A.I. Vogel. *A Text Book of Quantitative Inorganic Analysis*, 5th Edn, Longmans, London (1989).
- [19] D.C. Bradley, R.C. Mehrotra, F.M. Abd-el-Halim. *J. Chem. Soc.*, 4609 (1952).
- [20] J.P. Casey, K. Mislow. *J. Chem. Soc., Chem. Commun.*, 1410 (1970).
- [21] H.A. Meinema, J.G. Noltes. *J. Organomet. Chem.*, **25**, 139 (1970).
- [22] S. Bansal, Y.P. Singh, A. Singh. *Main Group Met. Chem.*, **28**, 149 (2005).
- [23] G.N. Chremos, R.A. Zingaro. *J. Organomet. Chem.*, **22**, 647 (1970).
- [24] R.G. Goyal, E. Maslowsky Jr, C.R. Senoff. *Inorg. Chem.*, **10**, 2572 (1971).
- [25] E. Maslowsky Jr. *J. Organomet. Chem.*, **70**, 153 (1974).
- [26] G.O. Doak, G.G. Long, L.D. Freedman. *J. Organomet. Chem.*, **4**, 82 (1965).
- [27] G.E. Maciel, J.J. Natterstad. *J. Chem. Phys.*, **42**, 2427 (1965).
- [28] G.N. Bodner, L.J. Todd. *Inorg. Chem.*, **13**, 360 (1974).
- [29] C. Eischenbroich, A. Salzer. *Organometallics: A Concise Introduction*, 2nd Edn, VCH Weinheim, New York (1992).
- [30] H.A. Meinema, J.G. Noltes. *J. Organomet. Chem.*, **107**, 249 (1976).
- [31] J.N.R. Ruddick, J.R. Sams. *J. Organomet. Chem.*, **128**, C41 (1977).
- [32] B.G. Tweedy. *Phytopathology*, **55**, 910 (1964).
- [33] S. Gaur, S. Manju, N. Fahmi, R.V. Singh. *Main Group Met. Chem.*, **28**, 293 (2005).
- [34] T.C. Sharma, V. Saxena, N.J. Reddy. *Acta Chim.*, **93**, 4 (1977).