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Antimony(III) and bismuth(III) complexes containing O- and N-chelating ligands: synthesis, FT-IR, ^{1}H and ^{13}C NMR spectroscopic and mass spectrometric studies

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Abstract: Reaction of $SbCl_3$ with sodium salt of vanillinidene-2-methylaminobenzene (vmab**H**) and salicylidene-3-nitroaminobenzene (snab**H**) in 1:1 and 1:2 molar ratios afforded complexes of the type $[Sb(\eta^2\text{-sb})_nCl_{3-n}]$(**1–4**) [where sb = vanillinidene-2-methyl-1-aminobenzene (vmab**H**) (**1, 2**) and salicylidene-3-nitroaminobenzene (snab**H**) (**3, 4**)]. Bismuth complexes of type $[Bi(PBT)_nCl_{3-n}]$ (**5, 6**) have been synthesized similarly by the reaction of $BiCl_3$ with sodium salt of 2-(*o*-hydroxyphenyl)-benzothiazole (**H**PBT) in different molar ratios (where n = 1–2). These newly synthesized complexes of antimony and bismuth have been characterized by elemental (C, H, N, S, Sb, and Bi) analysis and spectroscopic IR, ^{1}H NMR, and ^{13}C NMR techniques. The molecular composition of three selective complexes has been confirmed by mass spectrometric studies.

Keywords: antimony(III) complexes; bismuth(III) complexes; 2-(*o*-hydroxyphenyl)-benzothiazole (**H**PBT).

Introduction

Antimony and bismuth complexes derived from Schiff bases are given considerable importance due to their significant biological properties which lead to therapeutic effect (Tiekink, 2002; Ge and Sun, 2007). Antimony complexes have shown significant potential against tumor cell (Hadjikakou et al., 2015) and are used as antileishmanial (Khan et al., 2011), anthelminthic (Newlove et al., 2011), antitrypanosomal (Parrilha et al., 2012), antimicrobial (Kasuga et al., 2006), and anticancer (Ozturk et al., 2012) agents. Furthermore, antimony complexes have shown prominent application in catalysis (Ning et al., 2016). Bismuth(III) complexes are well acknowledged for their role in organic synthesis (Bothwell et al., 2011), nano material synthesis (Stavila et al., 2009), medicine (Briand and Burford, 1999), and biocoordination (Yang and Sun, 2007). Recently Bi(III) Schiff base complexes demonstrated strong inhibitory action against microbes (Li et al., 2015) and as antibiotics against *Helicobacter pylori* (Andrews et al., 2014; Pathak et al., 2015). Taking above facts in view, our interest in main group metal complexes (Dubey et al. 2011b, 2013, 2014) prompts us to synthesize antimony and bismuth complexes with biologically potent ligands such as 2-(*o*-hydroxyphenyl)-benzothiazole (**H**PBT) (Singh et al. 2014), which also is known for its electroluminescent properties (Yu et al., 2003) and Schiff bases (Dubey et al. 2011a, 2012, 2014) derived by condensation of *o*-vanillin, salicylaldehyde with *o*-toluidine, *m*-nitroaniline, respectively.

Results and discussion

The antimony(III) complexes **1–4** have been prepared by the reaction of antimony trichloride with sodium salt of vanillinidene-2-methylaminobenzene (vmab**H**) and salicylidene-3-nitroaminobenzene (snab**H**) in 1:1 and 1:2 ratios as shown in Scheme 1. Bismuth(III) complexes have been synthesized by the reaction of bismuth trichloride with sodium salt of 2-(*o*-hydroxyphenyl)-benzothiazole (**H**PBT) in different molar ratios as shown in Scheme 2. Antimony complexes are dark yellow to brown colored solids, whereas bismuth complexes are creamy white solid. These complexes are insoluble in organic solvents and soluble in polar solvents. Some physical properties of these complexes are summarized in Table 1.

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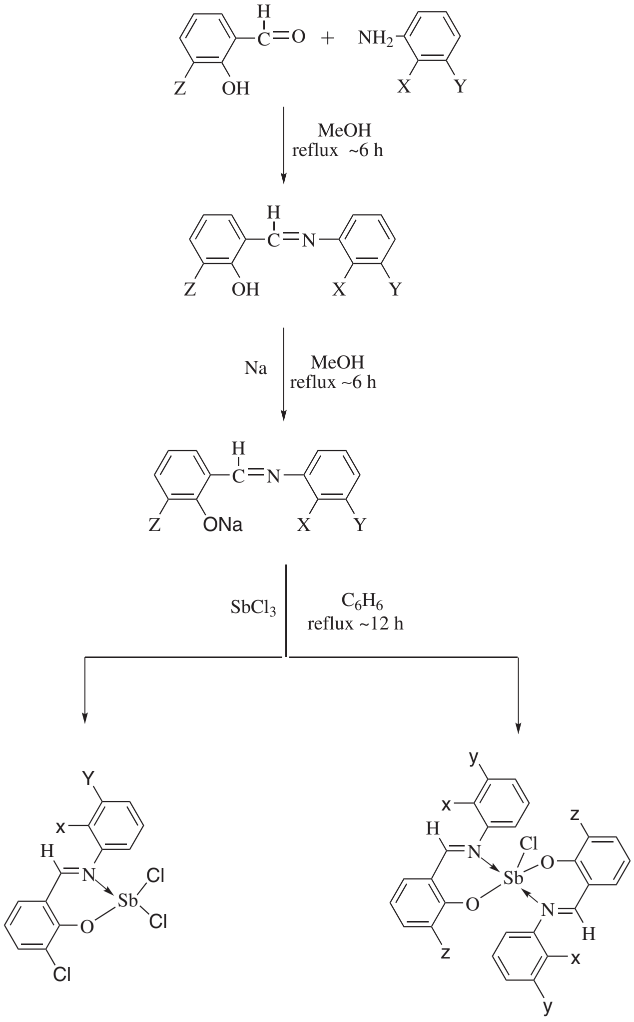


1, 3

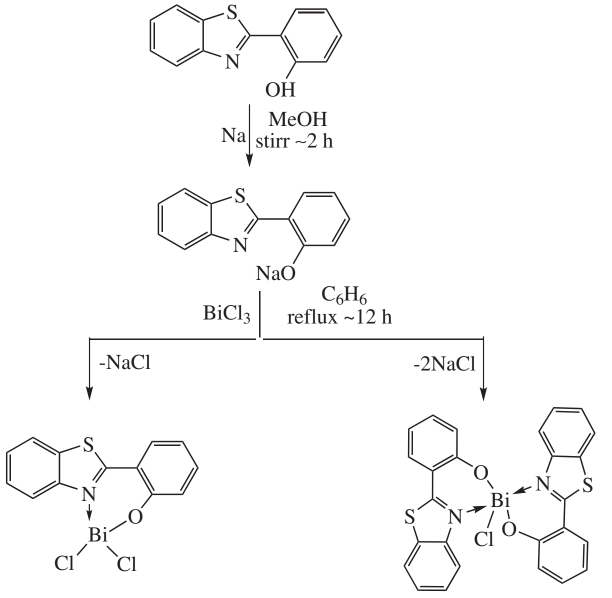
2, 4

Complex	X	Y	Z
1	CH_3	H	OCH_3
2	CH_3	H	OCH_3
3	H	NO_2	H
4	H	NO_2	H

Scheme 1: General method for the preparation of Schiff bases and antimony(III) complexes.

IR spectroscopy

Salient absorption bands were observed in IR spectra for complexes **1–6** and free ligands, which are summarized in Table 2. The characteristic feature of the IR spectra of the complexes is disappearance of phenolic stretching vibration $\nu_{(O\text{-}H)}$ observed in free ligands in the region 3564–3343 cm^{-1} due to deprotonation of phenolic hydrogen (Viswanathamurthi et al., 2000), which was further supported by shift of $\nu_{(C\text{-}O)}$ band ~10–20 cm^{-1} higher frequency region in complexes compared to free ligands in the region 1272–1295 cm^{-1} (Dubey and Singh, 2013) and appearance of new bands in the regions 573–580 and 546–570 cm^{-1} for bismuth and antimony complexes, respectively (Durgashanker and Singh, 2007). The involvement of azomethine nitrogen in bonding in complexes was supported by shifting of $\nu_{C=N}$ towards lower frequency 1615–1598 cm^{-1} region compared to free ligands 1634–1616 cm^{-1} region (Vajpayee and Singh, 2007) and appearance of new bands in the region 580–540 cm^{-1} due to M←N stretching vibration (Dawara and Singh, 2011).



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Scheme 2: General method for the preparation of bismuth(III) complexes.

^{1}H NMR spectroscopy

The ^{1}H NMR spectra recorded in DMSO-d$_6$ using tetramethylsilane as internal reference and ^{1}H NMR data of free ligands and their complexes **1–6** are collected in Table 3. A comparison of ^{1}H NMR spectra of ligand with metal complexes can lead to the following conclusions: (i) ^{1}H NMR spectra of complexes **1–6** exhibit no signal due to phenolic hydrogen (OH) observed at 13.20–12.76 ppm in the ligands, indicating metallation of phenolic group (OH) (Dubey et al., 2014); (ii) shifting of azomethine hydrogen signal to a higher region 9.93–8.32 ppm compared to parent Schiff bases (9.23–8.23 ppm) supporting

Table 1: Synthetic and analytical details of antimony(III) and bismuth(III) complexes **1–6**.

Complex empirical formula	Mol. wt.	Physical state	MP (°C)	Yield %	% Elemental analysis found (calcd)						
					C	H	N	S	Cl	Sb	Bi
vmab**H I**	241.29	Orange	95	78	74.25	6.40	5.69	–	–	–	–
$C_{15}H_{15}NO_2$		solid			(74.67)	(6.27)	(5.81)				
[Sb(η^2-vmab)Cl_2] **1**	432.94	Dark yellow	140	64	41.13	3.11	3.85	–	16.85	27.95	–
$C_{15}H_{14}Cl_2NO_2Sb$		solid			(41.61)	(3.26)	(3.24)		(16.38)	(28.12)	
[Sb(η^2-vmab)$_2$Cl] **2**	637.77	Dark yellow	132	60	56.48	4.63	4.20	–	5.55	18.87	–
$C_{30}H_{28}ClN_2O_4Sb$		solid			(56.50)	(4.43)	(4.39)		(5.56)	(19.09)	
snab**H II**	242.23	Yellow	105	82	64.12	4.29	11.23	–	–	–	–
$C_{13}H_{10}N_2O_3$		solid			(64.46)	(4.16)	(11.56)				
[Sb(η^2-snab)Cl_2] **3**	433.89	Brown	125	69	35.85	2.12	6.80	–	16.20	28.50	–
$C_{13}H_9Cl_2N_2O_3Sb$		solid			(35.99)	(2.09)	(6.46)		(16.34)	(28.06)	
[Sb(η^2-snab)$_2$Cl] **4**	639.66	Brown	148	62	48.75	2.55	8.75	–	5.62	19.10	–
$C_{26}H_{18}ClN_4O_6Sb$		solid			(48.82)	(2.84)	(8.76)		(5.54)	(19.04)	
HPBT **III**	227.28	White solid	130	–	68.65	3.75	6.25	14.00	–	–	–
$C_{13}H_9NOS$					(68.70)	(3.99)	(6.16)	(14.11)			
[Bi(PBT)Cl_2] **5**	506.16	Creamy	165	61	30.75	1.49	2.71	6.25	14.25	–	40.93
$C_{13}H_8BiCl_2NOS$		white solid			(30.82)	(1.59)	(2.77)	(6.33)	(14.01)		(41.29)
[Bi(PBT)$_2$Cl] **6**	696.98	Creamy	172	59	44.23	2.20	4.18	9.45	5.19	–	29.64
$C_{26}H_{16}BiClN_2O_2S_2$		white solid			(44.80)	(2.31)	(4.02)	(9.20)	(5.09)		(29.98)

Table 2: Characteristic IR frequencies (cm^{-1}) of new antimony(III) and bismuth(III) complexes **1–6**.

Complex	$\nu_{(OH)}$	$\nu_{(C=N)}$	$\nu_{(C\text{-}O)\ Phenolic}$	$\nu_{M\leftarrow N}$	$\nu_{(M\text{-}O)}$
vmab**H**	3564	1617	1272	–	–
[Sb(η^2-vmab)Cl_2] **1**	–	1608	1282	438	570
[Sb(η^2-vmab)$_2$Cl] **2**	–	1611	1284	438	580
snab**H**	3343	1614	1277	–	–
[Sb(η^2-snab)Cl_2] **3**	–	1609	1283	440	578
[Sb(η^2-snab)$_2$Cl] **4**	–	1607	1286	439	546
HPBT	3405	1640	1295	–	–
[Bi(PBT)Cl_2] **5**	–	1622	1315	455	573
[Bi(PBT)$_2$Cl] **6**	–	1620	1314	455	580

Table 3: ^{1}H NMR data (ppm) for new complexes **1–6**.

Complex	HC=N	Ar-H	Ar-OH	$-OCH_3$	$-CH_3$
vmab**H**	8.50 (s, 1H)	6.8–7.8 (m, 7H)	13.20 (s, 1H)	3.81 (s, 3H)	2.38 (s, 3H)
[Sb(η^2-vmab)Cl_2] **1**	9.66 (s, 1H)	7.8–8.0 (m, 7H)	–	3.89 (s, 3H)	2.40 (s, 3H)
[Sb(η^2-vmab)$_2$Cl] **2**	9.79 (s, 2H)	7.0–8.0 (m, 14H)	–	3.94 (s, 6H)	2.40 (s, 6H)
snab**H**	8.46 (s, 1H)	6.6–7.9 (m, 8H)	12.67 (s, 1H)	–	–
[Sb(η^2-snab)Cl_2] **3**	9.76 (s, 1H)	6.9–8.0 (m, 8H)	–	–	–
[Sb(η^2-snab)$_2$Cl] **4**	9.57 (s, 2H)	6.9–7.9 (m, 16H)	–	–	–
HPBT	8.23 (s, 1H)	6.8–7.8 (m, 8H)	11.74 (s, 1H)	–	–
[Bi(PBT)Cl_2] **5**	9.58 (s, 1H)	7.0–8.3 (m, 8H)	–	–	–
[Bi(PBT)$_2$Cl] **6**	9.65 (s, 2H)	7.0–8.3 (m, 16H)	–	–	–

coordination of azomethine nitrogen to metal atom (Solanki et al., 2009).

^{13}C NMR spectroscopy

The ^{13}C NMR data of free ligands and their complexes **1–6** are summarized in Table 4. In the ^{13}C NMR spectra of the Schiff bases, signals in the region 149.71–156.53 ppm due to azomethine carbon were shifted towards higher value in the region 152.16–159.19 ppm in the complexes, indicating the involvement of azomethine nitrogen

Table 4: ^{13}C NMR data (ppm) for new complexes of antimony(III) and bismuth(III) complexes.

Complex	C-O phenolic	HC = N	Ar-C	-OCH_3	Ar-CH_3
vamb**H**	145.8 (s, 1C)	151.6 (s, 1C)	132.5–122.7 (s, 11C)	54.1 (s, 1C)	18.1 (s, 1C)
[Sb(η^2-vmab)Cl_2] **1**	162.2 (s, 1C)	159.1 (s, 1C)	145.6–121.3 (s, 11C)	53.17 (s, 1C)	17.7 (s, 1C)
[Sb(η^2-vmab)$_2$Cl] **2**	159.1 (s, 2C)	155.5 (s, 2C)	147.7–121.8 (s, 22C)	54.07 (s, 2C)	17.5 (s, 2C)
snab**H**	144.4 (s, 1C)	149.7 (s, 1C)	147.4–123.2 (s, 11C)	–	–
[Sb(η^2-snab)Cl_2] **3**	161.1 (s, 1C)	152.1 (s, 1C)	143.2–121.9 (s, 11C)	–	–
[Sb(η^2-snab)$_2$Cl] **4**	157.1 (s, 2C)	157.3 (s, 2C)	139.4–120.6 (s, 22C)	–	–
HPBT	158.5 (s, 1C)	152.5 (s, 1C)	140.3–119.2 (s, 11C)	–	–
[Bi(PBT)Cl_2] **5**	163.4 (s, 1C)	154.5 (s, 1C)	135.5–119.5 (s, 11C)	–	–
[Bi(PBT)$_2$Cl] **6**	165.8 (s, 2C)	156.4 (s, 2C)	133.8–117.0 (s, 22C)	–	–

in bonding with antimony and bismuth atom (Dubey et al., 2014). Signal due to phenolic carbon in the region 144.45–156.53 ppm shifted toward the higher value in the region 157.19–165.81 ppm, indicating the involvement of phenolic oxygen in the bonding (Dubey et al., 2011b).

Mass spectrometry

The TOF-MS ES$^+$ spectra of the complexes **1**, **5**, and **6** were recorded. In the spectra of complexes **1**, **5**, and **6** molecular ion peak was observed at *m/z* 432.45 [($C_{15}H_{14}Cl_2NO_2Sb$); calculated mass = 430.94], 507.39 [($C_{13}H_8BiCl_2NOS$); calculated mass = 504.95] and 697.19 [($C_{26}H_{16}BiClN_2O_2S_2$); calculated mass = 696.01], respectively, which correspond to the monomeric molecular composition of the complexes.

Conclusion

Synthesis of antimony(III) and bismuth(III) complexes have been achieved by the reactions of $SbCl_3$ and $BiCl_3$ with appropriate sodium salt of ligands in 1:1 and 1:2 molar ratios, respectively. Spectroscopic studies confirm the O- and N-atom involvment in the bonding and molecular composition of complexes was proved by the mass spectrometric studies.

Experimental

Materials

$SbCl_3$ (Merck, Germany), $BiCl_3$, (*o*-hydroxyphenyl) benzothiazole (Sigma Aldrich, Bangalore, India), *o*-vanillin (Alfa-aesar), *o*-toluidine (LOBA, Mumbai, India), *m*-nitroaniline, and salicylaldehyde (CDH, New Delhi, India) were used without further purification. All solvents used were of reagent grade and purified by standard procedures (Armarego and Perrin, 1997). Chlorine was estimated by Volhard's method (Vogel, 2008) while antimony and bismuth were estimated complexometrically and iodometrically, respectively (Vogel, 2008). The Schiff bases were synthesized according to the literature method (Dubey et al., 2005).

Physical measurement

Elemental analyses for C, H, N, and S were carried out with Carlo Erba 1108 element analyzer. Melting points were measured in open tubes with a Stuart scientific apparatus and are uncorrected. Infrared spectra were recorded on a Perkin-Elmer 100 FT-IR spectrophotometer in the range 4000–400 cm^{-1}. ^{1}H and ^{13}C NMR spectra were recorded on Bruker Avance II 400 NMR spectrometer in DMSO-d_6 and chemical shifts were given in ppm relative to Me_4Si as internal reference. ESI-MS spectra were recorded on Agilent 6520 Q-Tof LC-MS, MS/MS spectrometers in acetonitrile.

Synthesis of complexes

Similar procedure was used for the preparation of the complexes (**1–6**); therefore, for the sake of brevity, general preparative details are given below for complex **1**. To a benzene (~20 mL) solution of $SbCl_3$ (1.140 g, 4.99 mmol) sodium salt of Schiff base [prepared by dissolving equimolar amount of sodium metal (0.114 g, 4.99 mmol) and a Schiff base, vanillinidene-2-methylaminobenzene (1.205 g, 4.99 mmol) in MeOH (~30 mL)] was added dropwise in 1:1 molar ratio with constant stirring. The reaction mixture was allowed to reflux for ~12 h. The precipitated NaCl was removed by filtration. The solvent was removed by distillation. The solid products were dried under reduced pressure and recrystallized from the mixture of THF- *n*–hexane in 1:4.

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