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SYNTHESIS, CHARACTERISATION AND REACTIONS OF CATIONIC COMPLEXES OF ARYLANTIMONY(III) CHLORIDES Ar_nSbCl_{3-n} WITH PERCHLORATE AND TETRAFLUOROBORATE ANIONS

Ram Nath Prasad Yadav

Department of Chemistry, Tribhuvan University, Thakur Ram Multiple Campus, Birganj, Nepal

E-mail: ramnathy@rocketmail.com

Abstract

Several hitherto unreported cationic complexes of the general formula $[Ar_2SbL][Y]$ and $[ArSbL_2][Y]_2$ [Where, Ar = C_6H_5, L = α-Picoline, Pyridine, Ph_3AsO, hexamethyl phosphoramide (HMPA), thiourea (TU) and Y = ClO_4^- BF_4^-] have been synthesized and characterized by solid state IR, ^{1}H NMR, elemental analysis, conductance and molecular weight measurements. The physico-chemical data, cations $[Ar_2SbL]^{+1}$ and$[ArSbL_2]^{+2}$ are assigned a pyramidal structure.

Key words: Arylantimony(III) chloride, Cationic complex, IR spectra, Elemental analysis, Molecular weight measurement, Perchlorate, Tetrafluoroborate.

Introduction

There has been a considerable interest centered around the chemistry of organoantimony(III) halides over the past two decades. As a result a variety of parent, $SbPh_nX_{3-n}$ (n = 1, 2) compounds where X = halides, imide, heterocyclic species, xanthates, thiophosphate, carboxylate have been synthesized and studies (Premraj *et al.* 1989^a, Premraj *et al.* 1989^b and Pandey 2001). Anionic complexes of pentafluorophenylantimony(III) chloride have been synthesized (Yadav 2012). Lewis acidity of $SbPh_nX_{3-n}$ (X = halide) towards monodentate and bidentate neutral O, N, and S donors and electronegatively charged donors has been investigated (Hall & Sowerby 1986 and Nunn *et al.* 1996). Apart from synthetic utility they have aroused interest due to their antitumour activity (Yadav 2012). A perusal of literature reveals that the existence and isolation of solid salts of complex cations of the type $[RSbL_2]^{+2}$ and $[R_2SbL]^+$ where L = monodentate neutral ligand in combination of an anionic group such as perchlorate, tetrafluoroborate has not been reported to date. In sharp contrast to this organoantimony(V) . cations of the type $[R_2SbL_3]^{+3}$ and $[R_3SbL_2]^{+2}$ are well documented (Nishi & Okawara 1972, Jha & Joshi 1984, Saxena *et al.* 1990, Premraj *et al* 1992, Singhal *et al.* 2003 and Singhal *et al.* 2004).

In the present communication we report the synthesis and reactions of two series of cationic complexes of the type $[Ar_2SbL][Y]$ and $[ArSbL_2][Y]_2$ where, Ar = Phenyl and L = α-picoline, pyridine, Ph_3AsO, HMPA and TU.

Solid state IR spectra and solution state ^{1}H NMR spectra added by some other solution phase studies have been carried out to characterize and elucidate the structure of newly synthesized compounds.

Result and Discussion

Under unhydrous oxygen free conditions, arylantimony(III) cationic complexes of the type $[Ar_nSbL_{3-n}][Y]_{3-n}$ can readily be obtained by the intraction of performed solution of arylantimony(III) chlorides, neutral monodentate ligand and silver and sodium salt of the anion in appropriate stoichiometry as shown below:

$$Ar_2SbCl + AgY/NaY \rightarrow [Ar_2SbL][Y] + AgCl/NaCl \qquad (1)$$

$$ArSbCl_2 + 2L + 2Na\ (or\ Ag)\ Y \rightarrow [ArSbL_2][Y]_2 + 2NaCl \qquad (2)$$

Where, Ar = C_6H_5 ; L = α-Picoline, Pyridine, Ph_3AsO, HMPA, TU; Y = ClO_4^- BF_4^-

All the reactions were found to proceed smoothly at room temperature affording excellent yields of the complexes. These compounds could be crystallised from petroleum/ether (40-60°C) solution and are obtained as white crystalline solids.

The consistency in melting points after repeated crystallization as well as mixed melting points rules out the possibility of the presence of unreacted reactants. The analytical data given in table 1–3 correspond well to the proposed formulation of the complexes. Conductance measurements values of 10^{-3} M solutions in acetonitrile for the complexes (**1, 2, 5, 13, 14, 16, 17, 18)** are in good agreement with the reported values of the 1:1 electrolytes in the solution. The conductance values are listed in Table-3.

IR spectra

An infrared spectrum for all the compounds was recorded in the range 4000-400 cm^{-1} in KBr or CsI pelets. The absorption frequencies inherent to phenyl groups are in close proximity reported for Ph_2SbCl and $PhSbCl_2$ (Hall & Sowerby 1986, Pandey 2001 and Singhal *et al.* 2003) and hence not discussed and tabulated. Diagnostic frequencies associated with ligands and characteristic anionic frequencies have been identified and discussed below.

Both pyridine and α-picoline exhibit characteristic vibration v(C=N), appearing in the range 1565-1580 cm^{-1} in free ligands, undergoes a positive shift of (1590-1610 cm^{-1}) suggesting coordination through nitrogen atom of the base (Premraj & Agarwal 1992 and Premraj *et al.* 1992).

In the case of TPAO (triphenyl arsenic oxide), the characteristics v(As-O), vibration is lowered in case of each complex as compared to its position in free ligand (880 cm^{-1}) and is shifted to 830-845 cm^{-1} (Premraj & Agarwal 1992 and Premraj *et al.* 1992).

The diagnostic v(P=O) vibration appearing at 1212 cm^{-1} in case of HMPA is considerably lowered 1130-1145 cm^{-1} on complexation. This lowering clearly, suggest coordination through nitrogen atom of the base (Premraj *et al.* 1989, Premraj & Agarwal 1992, Premraj *et al.* 1992 and Pandey 2001).

In case of thiourea a band of strong intensity appearing of 1045 ± 24 has been attributed to possess contribution from v(C=N) and v(C=S) modea. After complexation, v(C=N) exhibit a positive shift if the coordination takes place through the sulphur atom, while v(C=S) undergoes almost an equal negative shift. The resulting spectra of the complexes, therefore do not exhibit any peculiar change on coordination through sulphur atom and the resulting absorption remains apparently unchanged. The positive shift of v(N-H) from 3300 cm^{-1} in free thiourea to ~ 3370-3380 cm^{-1} in the complexes is suggestive coordination through nitrogen atom of the ligand (Premraj & Mishra 1991).

The v(Sb-O) was found in the range 415-435 cm^{-1} and is consistent with the earlier made assignment for complex having Sb-O linkage.

Due to the complexity of the spectra as well as limitation below 400 cm^{-1}, (Sb-N) stretching could not be assigned with certainty, v(Sb-N) band has reported to appear in the range 350-370 cm^{-1}.

The v(Sb-N) stretching corresponding to 'Y' mode of the phenyl group appear in the range 445-470 cm^{-1}.

The characteristic vibration due to BF_4^- and ClO_4^- do not show any significant shifting as compared to free anions vibration and given in Table – 4.

^{1}H NMR Spectra

The ^{1}H NMR spectra of two representative compound (**13 & 14)** showed singlet at 2.46 and 2.58 (due to methyl group) and multiplets for phenyl protons centered at δ 7.80 ppm, δ 7.25 ppm and δ 7.08 ppm of which the former (lower field multiplet) is attributed to ortho and the latter two (high field multiplet) are attributed to meta and para protons of phenyl groups.

Thus on the basis of IR, ^{1}H NMR spectra, molar conductance and molecular weight measurement, the newly synthesised complex cation are assigned a pyramidal structure as shown below:

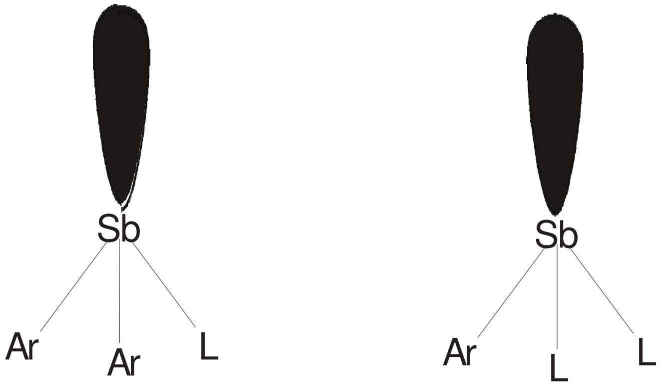

Fig: Suggested structures for $[Ar_2SbL]^{+1}$ and $[ArSbL_2]^{+2}$ cations.

Experimental

Diarylantimony(III) chloride, Ar_2SbCl and arylantimony(III) chlorides, ($ArSbCl_2$) were prepared by the addition of antimony trichloride to triphenylantimony. Anhydrous silver perchlorate was prepared by the standard procedure. Sodium tetrafluoroborate (Lobacheme) was used as such. All the ligands were of reagent grade and used without further purification. The solvents were purified dried before use. All manipulations were conducted in an atmosphere of nitrogen and stringent precautions were taken to moisture.

The melting points of the compounds were determined on the electrically operated melting point apparatus (M/s Toshniwal and Bros, Mumbai India).

The stoichiometry of the compound was established by elemental analysis. Percentage of C, H, and N the compounds was obtained on a semi-microscale (using elemental analysiser Carlo Eaba 1106, Thomas CH analyser and Coleman N analyser). Conductivity data were obtained in nitrobenzene with help of a Philips magic eye type PR 950 conductivity bridge using a dip type conductivity cell. IR spectra were recorded on a Perkin Elmer 577 spectrophotometer in the range 4000-200 cm^{-1} in KBr or CsI pellets. ^1H NMR spectra in $CDCl_3$ solution were determined on a Varian EM – 360 L spectrometer using TMS as an internal standard. Molecular weights data in benzene and nitrobenzene soluble compounds were determined cryoscopically in freezing benzene or nitrobenzene using a Beckman thermometer of accuracy ± 0.01°C. Typical experimental details of the few reactions are described below. Relevant IR assignments, ^1H NMR spectra, analytical data and molar conductance values are listed in table 1-4.

Reaction of Dphenylantimony(III) Chloride with α-picoline and Silver Perchlorate $[Ph_2Sb(\alpha\text{-pic})]ClO_4$ (1)

In an oxygen free environment diphenylantimony(III) chloride (0.311 g., 1 mmol) and neutral ligand α-picoline (0.093 g, 1 mmol) was taken in dry chloroform and silver perchlorate (0.207 g, 1 mmol) in dry methanol was added to this solution. After stirring the mixture for about 6 h; it was refluxed for 2 h and AgCl formed as a white precipitate was filtered off. The filtrate on concentration in *vacuo* afforded a white solid which was crystallized from petroleum ether (40-60 °C). M.P.: 160°C, Yield: 0.290 g, (62%).

Reaction of Phenylantimony(III) Dichloride with α-picoline and Sodium Tetrafluoroborate $[PhSb(\alpha\text{-pic})_2][BF_4]_2$ (4)

Phenylantimony(III) dichloride (0.270 g, 1 mmol) and neutral ligand α-picoline (0.186 g, 2 mmol) were taken in dry chloroform and sodium tetrafluoroborate (0.220 g, 2 mmol) in dry methanol was added to this solution. After stirring the mixture for about 5 h, it was then refluxed for 3 h and NaCl so formed as precipitate was filtered off. The filtrate on concentration in *vacuo*, afforded a light orange solid which was crystallized from petroleum ether (40-60°C). M.P.: 188°C, Yield: 0.419 g (75%).

Table 1: Preparation and properties of Arylantimony(III) cationic complexes

C. No.	Complexes	$Ar_nSbCl_{(3-n)}$ (g) n=1 & 2 Ag/Na Y (g) (solvent)	Ag/NaY (g) (solvent)	Ligand (g) (solvent)	M.P. (^oC)	Colour
	L=α-picoline					
1.	$[Ph_2SbL]$ $[ClO_4]$	Ph_2SbCl (0.311g) ($CHCl_3$)	$AgClO_4$ (0.207g) (MeOH)	α-Picoline (0.093g) ($CHCl_3$)	160	White
2.	$[Ph_2SbL]$ $[BF_4]$	Ph_2SbCl (0.311g) ($CHCl_3$)	$NaBF_4$ (0.110g) (MeOH)	α-Picoline (0.093g) ($CHCl_3$)	90	Off white
3.	$[PhSbL_2]$ $[ClO_4]_2$	$PhSbCl_2$ (0.270g) ($CHCl_3$)	$AgClO_4$ (0.415g) (MeOH)	α-Picoline (0.186g) ($CHCl_3$)	185	White
4.	$[PhSbL_2]$ $[BF_4]_2$	$PhSbCl_2$ (0.270g) ($CHCl_3$)	$NaBF_4$(0.220g) (MeOH)	α-Picoline (0.186g) ($CHCl_3$)	188	Light orange
	L=Pyridine					
5.	$[Ph_2SbL]$ $[ClO_4]$	Ph_2SbCl (0.311g) ($CHCl_3$)	$AgClO_4$ (0.207 g) (MeOH)	C_5H_5N (0.079 g) ($CHCl_3$)	143	White
6.	$[Ph_2SbL]$ $[BF_4]$	Ph_2SbCl (0.311g) ($CHCl_3$)	$NaBF_4$ (0.110 g) (MeOH)	C_5H_5N (0.079 g) ($CHCl_3$)	189	Cream
7.	$[PhSbL_2]$ $[ClO_4]_2$	$PhSbCl_2$ (0.270g) ($CHCl_3$)	$AgClO_4$ (0.415 g) (MeOH)	C_5H_5N (0.158 g) ($CHCl_3$)	190	White
8.	$[PhSbL_2]$ $[BF_4]_2$	Ph_2SbCl (0.311g) ($CHCl_3$)	$NaBF_4$ (0.220 g) (MeOH)	C_5H_5N (0.158 g) ($CHCl_3$)	168	Cream
	L=Ph_3AsO					
9.	$[Ph_2SbL]$ $[ClO_4]$	Ph_2SbCl (0.311g) ($CHCl_3$)	$AgClO_4$ (0.207 g) (MeOH)	Ph_3AsO (0.322g) ($CHCl_3$)	180	White
10.	$[Ph_2SbL]$ $[BF_4]$	Ph_2SbCl (0.311g) ($CHCl_3$)	$NaBF_4$ (0.110g) (MeOH)	Ph_3AsO (0.322g) ($CHCl_3$)	175	White
11.	$[PhSbL_2]$ $[ClO_4]_2$	$PhSbCl_2$ (0.270g) ($CHCl_3$)	$AgClO_4$ (0.415 g) (MeOH)	Ph_3AsO (0.644g) ($CHCl_3$)	185	White
12.	$[PhSbL_2]$ $[BF_4]_2$	Ph_2SbCl_2(0.270g)($CHCl_3$)	$NaBF_4$(0.220g) (MeOH)	Ph_3AsO (0.644) ($CHCl_3$)	190	White
13.	$[Ph_2SbL]$ $[ClO_4]$	Ph_2SbCl (0.311g) ($CHCl_3$)	$AgClO_4$ (0.207 g) (MeOH)	HMPA (0.179 g) ($CHCl_3$)	192	White
14.	$[Ph_2SbL]$ $[BF_4]$	Ph_2SbCl (0.311g) ($CHCl_3$)	$NaBF_4$ (0.110 g) (MeOH)	HMPA (0.179 g) ($CHCl_3$)	95	White
15.	$[PhSbL_2]$ $[ClO_4]_2$	$PhSbCl_2$ (0.270g) ($CHCl_3$)	$AgClO_4$ (0.415 g) (MeOH)	HMPA (0.358 g) ($CHCl_3$)	182	White
16.	$[PhSbL_2]$ $[BF_4]_2$	$PhSbCl_2$ (0.270g) ($CHCl_3$)	NaBF (0.220 g) (MeOH)	HMPA (0.358 g) ($CHCl_3$)	177	White
	L=Thiourea					
17.	$[Ph_2SbL]$ $[ClO_4]$	Ph_2SbCl (0.311g) ($CHCl_3$)	$AgClO_4$ (0.207 g) (MeOH)	H_2NCSNH_2 (0.076g) ($CHCl_3$)	174	Yellow
18.	$[Ph_2SbL]$ $[BF_4]$	Ph_2SbCl (0.311g) ($CHCl_3$)	$NaBF_4$ (0.110 g) (MeOH)	H_2NCSNH_2 (0.076g) ($CHCl_3$)	170	White
19.	$[PhSbL_2]$ $[ClO_4]_2$	$PhSbCl_2$ (0.270g) ($CHCl_3$)	$AgClO_4$ (0.415g) (MeOH)	H_2NCSNH_2 (0.152g) ($CHCl_3$)	182	White
20.	$[PhSbL_2]$ $[BF_4]_2$	$PhSbCl_2$ (0.270g) ($CHCl_3$)	$NaBF_4$ (0.220 g) (MeOH)	H_2NCSNH_2 (0.152g) ($CHCl_3$)	190	White

Table 2: Elemental analysis of Arylantimony(III) cationic complexes

C. No.	Complexes	Empirical formula	Found (Calcd) %		
			C	H	N
1.	[Ph_2SbL] [ClO_4]	$C_{18}H_{17}NClO_4Sb$	44.13 (46.13)	2.63 (3.63)	1.75 (2.09)
2.	[Ph_2SbL] [BF_4]	$C_{18}H_{17}NBF_4Sb$	46.41 (47.41)	2.73 (3.73)	2.97 (3.07)
3.	[$PhSbL_2$] [ClO_4]$_2$	$C_{18}H_{19}NCl_2O_8Sb$	36.0 (37.01)	2.26 (3.26)	3.40 (4.80)
4.	[$PhSbL_2$] [BF_4]$_2$	$C_{18}H_{19}N_2B_2F_8Sb$	36.65 (38.68)	2.10 (3.40)	4.00 (5.01)
5.	[Ph_2SbL] [ClO_4]	$C_{17}H_{15}NClO_4Sb$	42.91 (44.91)	22.00 (3.30)	2.01 (3.08)
6.	[Ph_2SbL] [BF_4]	$C_{17}H_{15}NBF_4Sb$	45.20 (46.20)	2.20 (3.40)	2.20 (3.17)
7.	[$PhSbL_2$] [ClO_4]$_2$	$C_{16}H_{15}N_2Cl_2O_8Sb$	33.55 (34.55)	1.70 (2.70)	4.04 (4.04)
8.	[$PhSbL_2$] [BF_4]$_2$	$C_{16}H_{15}N_2B_2F_8Sb$	35.20 (36.20)	1.83 (2.83)	4.28 (5.28)
9.	[Ph_2SbL] [ClO_4]	$C_{30}H_{25}ClO_5AsSb$	50.60 (5.64)	2.40 (3.59)	-
10.	[Ph_2SbL] [BF_4]	$C_{30}H_{25}BF_4OAsSb$	50.59 (52.59)	2.65 (3.65)	-
11.	[$PhSbL_2$] [ClO_4]$_2$	$C_{18}H_{35}Cl_2O_{10}As_2Sb$	26.60 (28.6)	3.10 (4.65)	-
12.	[$PhSbL_2$] [BF_4]$_2$	$C_{18}H_{35}B_2F_8O_2As_2Sb$	27.65 (29.66)	2.98 (4.81)	-
13.	[Ph_2SbL] [ClO_4]	$C_{18}H_{28}N_3ClO_5PSb$	37.98 (38.98)	4.05 (5.05)	6.58 (7.58)
14.	[Ph_2SbL] [BF_4]	$C_{18}H_{28}N_3BF_4OPSb$	37.88 (39.88)	3.17 (5.17)	5.76 (7.76)
15.	[$PhSbL_2$] [ClO_4]$_2$	$C_{18}H_{41}N_6Cl_2O_{10}P_2Sb$	27.40 (28.58)	4.30 (5.43)	10.12 (11.12)
16.	[$PhSbL_2$] [BF_4]$_2$	$C_{18}H_{41}N_6B_2F_8O_2P_2Sb$	28.10 (29.57)	4.00 (5.61)	10.21 (11.50)
17.	[Ph_2SbL] [ClO_4]	$C_{13}H_{14}N_2ClO_4Sb$	33.29 (34.57)	2.06 (3.10)	5.02 (6.21)
18.	[Ph_2SbL] [BF_4]	$C_{13}H_{14}N_2BF_4SSb$	34.57 (35.57)	2.19 (3.19)	5.38 (6.38)
19.	[$PhSbL_2$] [ClO_4]$_2$	$C_8H_{13}N_4Cl_2O_8S_2Sb$	16.40 (17.47)	1.31 (2.37)	9.10 (10.19)
20.	[$PhSbL_2$] [BF_4]$_2$	$C_8H_{13}N_4B_2F_8S_2Sb$	16.31 (18.31)	1.00 (2.48)	9.68 (10.68)

Reaction of Diphenylantimony(III) Chloride with Pyridine and Sodium Tetrafluoroborate [$Ph_2Sb.Py$][BF_4] (6)

In an anhydrous condition, diphenylantimony(III) chloride (0.311 g, 1 mmol) was taken in dry chloroform and sodium terafluoroborate (0.110 g, 1 mmol) in dry methanol was added to this solution. After stirring the mixture for about 6 h it was further refluxed for 3 h and NaCl so formed as a precipitate was filtered off. The filtrate on concentration in *vacuo*, afforded a cream colored solid which was crystallized from petroleum ether (40-60^{0}C). M.P.: 189^{o}C, Yield: 0.305 g, (69%).

Reaction of Phenylantimony(III) Dichloride with Triphenylarsine Oxide and Silver Perchlorate [$PhSb.(Ph_3AsO)_2$][ClO_4]$_2$ (11)

In an oxygen free environment, phenylantimony(III) dichloride (0.270 g, 1 mmol) and neutral ligand triphenylarsine oxide (0.644 g, 2 mmol) were taken in dry chloroform and silver perchlorate (0.415 g, 2 mmol) in dry methanol was added to this solution. After stirring the mixture for about 6 h, it was further refluxed for 3 h and AgCl so formed as a precipitate was filtered off. The filtrate on concentration in *vacuo,* afforded a white solid which was crystallized from petroleum ether (40-60^{o}C). M.P.: 185^{o}C, Yield: 0.558 g, (74%).

Table-3: Molar conductance and yield of Arylantimony(III) cationic complexes

C. No.	**Complex**	**Molar Conductance in Nitrobenzene (Ohm^{-1} cm^2 mol^{-1})**	**Yield**	
			(g)	**(%)**
1.	$[Ph_2SbL]$ $[ClO_4]$	52.4	0.290	62
2.	$[Ph_2SbL]$ $[BF_4]$	54.2	0.305	67
3.	$[PhSbL_2]$ $[ClO_4]_2$	50.6	0.409	70
4.	$[PhSbL_2]$ $[BF_4]_2$	51.7	0.419	75
5.	$[Ph_2SbL]$ $[ClO_4]$	56.2	0.359	79
6.	$[Ph_2SbL]$ $[BF_4]$	59.2	0.305	69
7.	$[PhSbL_2]$ $[ClO_4]_2$	56.2	0.411	74
8.	$[PhSbL_2]$ $[BF_4]_2$	50.7	0.424	80
9.	$[Ph_2SbL]$ $[ClO_4]$	53.4	0.411	59
10.	$[Ph_2SbL]$ $[BF_4]$	51.9	0.411	60
11.	$[PhSbL_2]$ $[ClO_4]_2$	58.3	0.558	74
12.	$[PhSbL_2]$ $[BF_4]_2$	57.6	0.553	76
13.	$[Ph_2SbL]$ $[ClO_4]$	59.5	0.300	54
14.	$[Ph_2SbL]$ $[BF_4]$	54.7	0.379	70
15.	$[PhSbL_2]$ $[ClO_4]$	57.6	0.544	72
16.	$[PhSbL_2]$ $[BF_4]_2$	51.5	0.577	79
17.	$[Ph_2SbL]$ $[ClO_4]$	58.3	0.343	76
18.	$[Ph_2SbL]$ $[BF_4]$	53.9	0.290	66
19.	$[PhSbL_2]$ $[ClO_4]_2$	51.8	0.379	69
20.	$[PhSbL_2]$ $[BF_4]_2$	54.2	0.378	72

Table 4: IR data of Arylantimony (III) cationic complexes

Comp. No.	ν(C=N)/ν(N-H) Complex	ν(As-O)/ ν(P-O) Free	Δν	νSb-C	Anion Frequencies
1	1590	1575	15	445	1990 vs 620s
2	1600	1575	25	450	1018 vs 520s
3	1595	1575	20	447	1990 vs 620s
4	1600	1575	25	460	1018 vs 520s
5	1598	1575	23	465	1990 vs 620s
6	1602	1575	27	450	1015 vs 520s
7	1590	1575	15	471	1990 vs 620s
8	1611	1575	36	765	1018 vs 520s
9	830	880	50	762	1920 vs 620s
10	835	880	45	745	1018 vs 520s
11	840	880	40	450	1990 vs 620s
12	845	880	35	465	1018 vs 520s
13	1130	1211	81	470	1990 vs 620s
14	1134	1212	78	404	1018 vs 526s
15	1140	1212	72	450	1990 vs 620s
16	1146	1212	66	440	1018 vs 520s
17	3370	3300	70	445	1990 vs 620s
18	3374	3300	74	455	1018 vs 520s
19	3376	3300	76	460	1990 vs 620s
20	3380	3300	80	470	1018 vs 520s

vs = very strong, s = strong.

Reaction of Diphenylantimony(III) Chloride with HMPA and Silver Perchlorate $[Ph_2Sb.HMPA][ClO_4]$ (13)

In an anhydrous condition, diphenylantimony(III) chloride (0.311 g, 1 mmol) and neutral ligand HMPA (0.179 g, 1 mmol) was taken in dry chloroform and silver perchlorate (0.207 g, 1 mmol) in methanol was added to this solution. After stirring the mixture for 7 h, it was refluxed for 2 h and AgCl so formed as a precipitate was filtered off. The filtrate on concentration in *vacuo,* afforded a white solid which was crystallized from petroleum ether (40-60^{0}C). M.P.: 192^{0}C), Yield: 0.300 g, (54%).

Reaction of Phenylantimony(III) Dichloride with Thiourea and Sodium Terafluoroborate $[PhSb.(TU)_2][BF_4]_2$ (20)

In an oxygen free environment, phenylantimony(III) dichloride (0.270 g, 1 mmol) and neutral ligand thiourea (0.152 g, 2 mmol) were taken in dry chloroform and sodium tetrafluoroborate (0.220 g, 2mmol) in dry methanol was added to this solution. After stirtring the mixture for 7 h, it was refluxed for 2 h and NaCl formed so as a precipitate was filtered off. The filtrate on concentration in *vacuo,* afforded a white solid which was crystallized from benzene. M.P.: 190^{0}C, Yield: 0.378 g, (72%).

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