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## PREPARATION AND CHARACTERISATION OF COMPLEX BIS(PENTAFLUOROPHENYL) ANTIMONY(V) ANIONS OF THE TYPE $[(R_f)_2SbCl_3X]^-$

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### Abstract

A series of hitherto unreported solid salts of hexa-coordinated anions of the general formula  $[(R_f)_2SbL_3X]^-$ , where  $R_f = C_6F_5$ ;  $L = Cl$  and  $X = Cl, Br, I, N_3, NCS$  and  $SeCN$  have been prepared in the presence of tetraorgano ammonium, phosphonium, arsonium and stibonium counter ions. The complexes have been formulated and characterised on the basis of elemental analysis, molar conductances, molecular weights and solid state IR, solution state  $^1H$  NMR and  $^{19}F$  NMR spectral data. The physico-chemical data are consistent with six-coordinate antimony complexes.

**Key words:** Bis(pentafluorophenyl)antimony(V) chloride, Anionic complex,  $^{19}F$  NMR spectra, Ammonium, Phosphonium, Arsonium, Stibonium.

### Introduction

Diorgano antimony(V) halides of the type  $R_2SbX_3$  ( $R = C_6H_5, C_6F_5, CH_3$ ) act as acceptor and form molecular adducts with monodentate Lewis bases extending the coordination number upto 6 (Nishii *et al.*, 1969, Jha & Joshi 1984 and Premraj & Mishra 1991). Hexacoordinate complexes of  $Ar_2SbCl_3$  and  $M_2SbCl_3$  with Schiff's bases, containing oxinate and acetyl acetonate groups have also been reported (Meinema *et al.* 1969, Meinema *et al.* 1972, Meinema & Noltes 1976 and Saxena *et al.* 1990). Unlike their hydrocarbon counterpart  $(C_6F_5)_2SbCl_3$  extend coordination number upto 7 having  $sp^3d^3$  hybridisation around antimony (Saxena *et al.* 1990). This may be ascribed to enhanced Lewis acidity of  $(C_6F_5)_2SbCl_3$  compared to diphenylantimonytrichloride due to the electron withdrawing nature of pentafluorophenyl group. Diorganoantimony(V) chloride also known to form complex anions  $[R_2SbX_3Y]^-$  by accepting an electronegative moiety *viz.*, halide and pseudohalide *etc.* (Bhattacharya *et al.* 1980). Among such classes of complexes, Bertazzi *et al.* reported the synthesis and isolation of mixed chlorohalogeno and mixed chloropseudohalogenodiphenyl antimonates(V) of the type  $[R_4M] [R_2SbX]$  and  $[R_4M] [R_2SbX_3Y]$  ( $M = N, As$ ;  $X = Y = Cl, Br, N_3, NCS$ ) (Bertazzi 1976). A similar class of complexes,  $[Me_4M] [cis-R_2SbCl_4]$  having hexa-coordinate antimony(V) species and where Sb-C bonds are forced into *cis* positions have

been reported by Meinema *et al.* (Meinema *et al.* 1977). An octahedral structure with trans organic groups has been deduced for  $[Me_2SbCl_4]^-$  with the aid of IR and Raman spectra. A few diarylantimonates(V) anionic complexes have been prepared and isolated with tetraalkyl ammonium, phosphonium and stibonium cations (Pandey 2001).

Anionic complexes of diarylantimony trichloride in combination of some unsymmetrical tetraorganostibonium salts preferably having one or more pentafluorophenyl group have been isolated by Firoz Zee (Zee 1990). The introduction of pentafluorophenyl group on to the antimony atom has been reported to render thermal stability as well as water solubility to such complexes. Only recently solid complex cation in combination with tetrafluoroborate, tetrafluorophenylborate and silver perchlorate of the general formula  $[R_2SbL_3]Y_3$  have been isolated in this laboratory (Sinhgal *et al.* 2003). The synthesis of diarylantimonates of the type  $[Ar_2SbCl_3X]$  isolated in combination of symmetrical tetraammonium and stibonium cations,  $R_4M^+[M = N, Sb]$  has also been carried out (Pandey 2001).

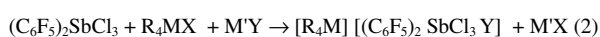
Thus the studies on bis(pentafluorophenyl)antimony(V) trichlorides seem to be confined to the synthesis of neutral and cationic complexes. No complex anion of the type  $[(C_6F_5)_2SbCl_3X]^-$  has been isolated to date. In sharp contrast to this pentafluorophenylantimony(III) anions of the type  $[(C_6F_5)_2SbClX]^-$ ,  $[(C_6F_5)_2SbCl_2X]^-$

and  $[\text{C}_6\text{F}_5\text{SbCl}_2]^{-2}$  and arylantimony(III) cations of the type  $[\text{Ar}_2\text{SbL}]^{+1}$  and  $[\text{ArSbL}_2]^{+2}$  are well documented (Yadav 2012 and Yadav 2013). In the present investigation the author has synthesized the series of hitherto unreported anionic complexes of the general formula  $[(\text{C}_6\text{F}_5)_2\text{SbCl}_3\text{X}]^-$  ( $\text{X}=\text{Cl}, \text{Br}, \text{I}, \text{N}_3, \text{NCS}$  and  $\text{SeCN}$ ), the objectives of this studies were two folds (i) isolation and characterisation of anionic antimonates having pentafluorophenyl group and to explore the possibility of the coordination number of antimony being extended beyond 6 or not (ii) and to establish the mode of bonding of pseudohalide group to antimony bearing pentafluorophenyl groups.

About the dozen of new anionic complexes have been isolated and characterised with the aid of elemental analysis, molecular weight and conductivity data. Vibration studies along with solution studies supplementary to above have been made to elucidate the structure of the newly synthesized compounds.

## Results and Discussion

Anionic complexes of bis(pentafluorophenyl)antimony trichloride in combination of symmetrical tetraorgano-ammonium, -phosphonium, -arsonium and -stibonium salts can be obtained by either of the two methods.



Where,  $\text{M} = \text{N}, \text{P}, \text{As}, \text{Sb}$ ;  $\text{X} = \text{Cl}, \text{Br}, \text{I}, \text{N}_3, \text{NCS}, \text{SeCN}$ ;  $\text{M}' = \text{Na}, \text{K}, \text{NH}_4^+$ ;  $\text{Y} = \text{N}_3, \text{NCS}, \text{SeCN}$ ;  $\text{R} = \text{CH}_3, \text{C}_4\text{H}_9, \text{C}_6\text{H}_5$ .

## Infrared spectra

Infrared spectra of all the complexes was recorded in the range  $4000\text{-}400\text{ cm}^{-1}$ . All the compounds show IR absorptions bands characteristics of the  $\text{C}_6\text{F}_5$  groups absorptions corresponding to the aryl and alkyl group in onium salts and are in close proximity reported for such compounds as well. The non mass sensitive vibration are therefore excluded from the discussion. Among the six mass sensitive modes, vibration corresponding to antimony-carbon mode assignable to  $\nu(\text{Sb-C})$  represented by  $\gamma$ -mode has been identified. The assignment of the peaks is tentative due to the overlapping and complex nature of the spectra owing to the presence of various organic groups.

In case of the bis(pentafluorophenyl)antimony(V) anions having azido group ( $-\text{N}_3$ ); the most prominent absorption in the infrared spectra were observed in the range  $2075\pm 10\text{ cm}^{-1}$  and is assignable to asymmetric stretching frequency. A weak absorption around  $1264\pm 6\text{ cm}^{-1}$  is attributed to symmetric mode. The position of asymmetric band is more in favour of covalently

The complexes were synthesized by the direct interaction of  $(\text{C}_6\text{F}_5)_2\text{SbCl}_3$  with tetraorgano-ammonium, -stibonium and -arsonium halides or pseudohalides. Mixed halo, pseudohalo complex anions could also be obtained by the interaction of  $(\text{C}_6\text{F}_5)_2\text{SbCl}_3$  with tetraorgano salts in presence of alkali metal or ammonium pseudohalides. The complexes were obtained by using the equimolar amounts of the reactants.

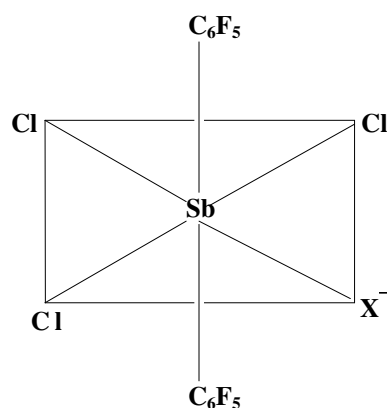
The reactions are quantitative and proceed smoothly in mild conditions. The constancy in the melting points of the complexes after repeated crystallisation excluded any possibility of the presence of mixture of reactants. The formula assigned to the complexes reported herein are consistent with the experimental data.

The complexes are soluble in ethanol, chloroform, acetonitrile, nitrobenzene and dichloromethane. They are off white solids and quite stable at room temperature and can be stored for several weeks without decomposition.

The molar conductance values for all the complexes were found to be in the range  $120.4\text{-}132.2$  in acetonitrile and in methanol, in the range  $108\text{-}123\text{ Ohm}^{-1}\text{ mole}^{-1}\text{ cm}^2$  which suggest them to be 1:1 electrolyte in the solvent and confirms the presence of two ions in the solution (Table 3). The value of Van't Hoff factor(i) was found to be between 1.76 - 2.30 which indicates the presence of two ions in the solution.

Diagnostic infrared absorption of pseudohalide groups together with their assignments are listed in Table 4. In the anionic complexes having thiocyanate group, the possibility of the group being nitrogen or sulphur bonded can readily be distinguished by the appearance of strong asymmetric stretching band at  $2010\pm 10\text{ cm}^{-1}$  suggesting it to be N-bonded. The presence of the Sb-N bond can further be ascertained by the bonding mode ( $\delta$ ) of NCS group appearing in the range  $460\text{-}470\text{ cm}^{-1}$ . A weak band around  $860\pm 5\text{ cm}^{-1}$  can be assigned to the symmetric mode of the isothiocyanate group. The observed frequencies are thus consistent with iso structural compounds containing Sb-NCS bonding (Bertazzi *et al.* 1974, Bertazzi *et al.* 1975 and Bertazzi 1976).

bonded azido ( $-\text{N}_3$ ) group attached to central antimony atom. The bending mode of vibration due to azido group ( $-\text{N}_3$ ) was observed around  $648\pm 7\text{ cm}^{-1}$ . The position of this band suggest the presence of covalently bonded linear azido group ( $-\text{N}_3$ ) (Bhattacharya *et al.* 1980).



X	Compound
I	(1)
I	(2)
Cl	(3)
Br	(4)
NCS	(5)
N <sub>3</sub>	(6)
Br	(7)
Br	(8)
NCS	(9)
N <sub>3</sub>	(10)
SeCN	(11)

Fig 1: Suggested structure of  $[(C_6F_5)_2SbCl_3X]^-$  complex anion.

Like the above two pseudohalides, IR spectra has been frequently used as an important aid to establish the distinction between the two isomeric selenocyanate (SeCN) forms. Like thiocyanates selenocyanates are also capable of exhibiting isomerism and can be either in the normal or the iso-form. They give rise to three fundamental modes of vibration in their IR spectra, two of which are  $\nu C=N$  and  $\nu C-Se$  while the third is due to a deformation mode of  $\delta NCS$ . The observations made so far by the various groups of workers indicate that the position and intensity of the peaks vary with the nature of metal.

The presence of sharp band of strong intensity around  $2065\text{ cm}^{-1}$  can be assigned to asymmetric NCS group. The observed frequency is consistent to the iso structure and is consistent with the usual iso structure of the thiocyanate counter part of the antimony(V) derivatives. Apart from this an absorption of weak intensity around  $630\text{ cm}^{-1}$  also lends support to the iso structure of the newly synthesized compound (Premraj *et al.* 1986).

The deformation mode corresponding to  $\delta NCS$  could not be assigned with certainty due to the overlapping of antimony-carbon stretching corresponding to Y mode in the same region. It may be noted that unlike the halogen and pseudohalogen vibration the exact identification of the various M-C vibrations are not certain as they exist both in the anion in the form of  $[(C_6F_5)_2SbCl_3X]^-$  or  $R_4E^+$  in the cation. This difficulty was however, overcome by Italian workers by

incorporating some Raman data as well and from the number of Raman and IR coincidences and assigned trans phenyl arrangement around the central atom of the anion for  $[Ph_2SbCl_4]^-$  anions (Bertazzi *et al.* 1974 and Bertazzi 1976). Under the similar conditions an octahedral environment with trans pentafluorophenyl is tentatively assigned to the anions reported in this work. However, considerable distortion from an ideal structure can not be ruled out due to the presence of different anions.

### NMR spectra

$^1H$  NMR spectra of compound (2) was recorded in  $CDCl_3$  using TMS as an internal reference. The appearance of a singlet  $\delta 2.80$  ppm for methyl protons. Indicate that all the four methyl groups are magnetically equivalent at room temperature and thus seemed to be in one plane.

The  $^{19}F$  NMR spectrum of the compound (2) was recorded in  $CDCl_3$  using  $CF_3COOH$  as reference at 85.26 MHz. The characteristic signals of  $F_{3,5}$  and  $F_4$  were observed at  $\delta$ -130.5 to -127.6 (d) ppm; -154.4 to -158.4 (d) ppm and -144.8 to -146.2 to -142.4 (t) ppm respectively. The peak of  $F_4$  was easily recognized due to half intensity compared to  $F_{2,6}$  and  $F_{3,5}$  peaks. The  $F_4$  signals appeared as triplet of triplet due to spin-spin coupling with  $F_{3,5}$ .

Thus on the basis of IR,  $^1\text{H}$  and  $^{19}\text{F}$  NMR spectra and conductance measurements, the newly synthesised complex anions  $[(\text{C}_6\text{F}_5)_2\text{SbCl}_3\text{X}]^-$  may be assigned an octahedral structure in which four ligands are situated at the corner positions while the pentafluorophenyl groups are settled in apical positions with C-Sb-C skeleton. Similar structure has earlier been assigned by other workers as well (Bertazzi 1976 and Bhattacharya *et al.* 1980).

### Experimental

Bis(pentafluorophenyl)antimony(V) chloride was prepared by the addition of dry chlorine to bis(pentafluorophenyl)antimony(III) chloride. Tetraphenylstibonium bromide, tetraphenylstibonium isothiocyanate, tetraphenylstibonium azide, tetraphenylstibonium selenocyanate, tetraphenylphosphonium bromide, tetraphenylarsonium isothiocyanate and tetraphenylarsonium azide were prepared by the standard method. Tetrabutylammonium iodide and tetramethylammonium chloride were of reagent grade and used without further purification. The solvents were purified and dried before use. All manipulations were conducted in an atmosphere of nitrogen and stringent precautions were taken to exclude moisture.

Conductivity data were obtained in acetonitrile and methanol 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  $4000\text{--}200\text{ cm}^{-1}$  in KBr or CsI pellets.  $^1\text{H}$  NMR spectra in  $\text{CDCl}_3$  solution were determined on a Varian EM – 360 L spectrometer using TMS as an internal standard. Typical experimental details of the few reactions are described below. Analytical data, molar conductance values and IR assignments are listed in table 1-4

#### Preparation of $[(\text{C}_4\text{H}_9)_4\text{N}][(\text{C}_6\text{F}_5)_2\text{SbCl}_3\text{I}]$ (1)

$(\text{C}_4\text{H}_9)_4\text{NI}$  (3.69 g, 0.01 mol) and  $(\text{C}_6\text{F}_5)_2\text{SbCl}_3$  (5.62 g, 0.01 mol) were refluxed in methanol (60 ml) in 1:1 molar ratio for 3 hrs. The resulting solution was filtered, concentrated and cooled over night to afford a crystalline compound. It was recrystallized from methanol and diethyl ether mixture (1:1). M.P.:  $170^\circ\text{C}$ , Yield: 0.745 g, (80%).

#### Preparation of $[(\text{CH}_3)_4\text{N}][(\text{C}_6\text{F}_5)_2\text{SbCl}_3]$ (3)

$(\text{CH}_3)_4\text{NCl}$  (1.09g; 0.01 mol) and  $(\text{C}_6\text{F}_5)_2\text{SbCl}_3$  (5.62 g, 0.01 mol) were refluxed in methanol (60 ml) in 1:1

molar ratio for 3 hrs. The resulting solution was filtered, concentrated and cooled over night to afford a crystalline compound. It was recrystallized from methanol and diethyl ether mixture (1:1). M.P.:  $140^\circ\text{C}$ , Yield: 0.477 g, (71%).

#### Preparation of $[(\text{C}_6\text{H}_5)_4\text{Sb}][(\text{C}_6\text{F}_5)_2\text{SbCl}_3\text{Br}]$ (4)

$(\text{C}_6\text{H}_5)_4\text{SbBr}$  (5.10 g, 0.01 mol) and  $(\text{C}_6\text{F}_5)_2\text{SbCl}_3$  (5.62 gm, 0.01 mol) in 1:1 molar ratio were refluxed in methanol for a period of 3 hrs. The solution was concentrated and cooled over night in a freeze to afford a solid which was crystallized with methanol and diethyl ether mixture (1:1). M.P.:  $125^\circ\text{C}$ , Yield: 0.750 g, (70%).

#### Preparation of $[(\text{C}_6\text{H}_5)_4\text{Sb}][(\text{C}_6\text{F}_5)_2\text{SbCl}_3\text{NCS}]$ (5)

$(\text{C}_6\text{H}_5)_4\text{SbNCS}$  (4.88 g; 0.1 mol, and  $(\text{C}_6\text{F}_5)_2\text{SbCl}_3$  (5.62 g 0.01 mol) were refluxed in methanol (60 ml) in 1:1 molar ratio for 3 hrs. The resulting solution was filtered, concentrated and cooled over night to afford a crystalline compound. It was recrystallized from methanol and diethyl ether mixtures (1:1). M.P.:  $165^\circ\text{C}$ , Yield: 0.787 g, (75%).

#### Preparation of $[(\text{C}_6\text{H}_5)_4\text{Sb}][(\text{C}_6\text{F}_5)_2\text{SbCl}_3\text{N}_3]$ (6)

$(\text{C}_6\text{H}_5)_4\text{SbN}_3$  (4.72 g; 0.01 mol) and  $(\text{C}_6\text{F}_5)_2\text{SbCl}_3$  (5.62; 0.01 mol) were refluxed in methanol (60 ml) in 1:1 molar ratio for 3 hrs. The resulting solution was filtered, concentrated and cooled over night to afford a crystalline compound. It was recrystallized from methanol and diethyl ether mixture (1:1). M.P.:  $180^\circ\text{C}$ , Yield: 0.744 g, (72%).

#### Preparation of $[(\text{C}_6\text{H}_5)_4\text{P}][(\text{C}_6\text{F}_5)_2\text{SbCl}_3\text{Br}]$ (8)

$(\text{C}_6\text{H}_5)_4\text{PBr}$  (4.19 g; 0.01 mol) and  $(\text{C}_6\text{F}_5)_2\text{SbCl}_3$  (5.62 g; 0.01 mol) in 1:1 molar ratio were refluxed in methanol for a period of 3 hrs. The solution was concentrated and cooled over night in a freeze to afford a solid which was crystallized with methanol and diethyl ether mixture (1:1). M.P.:  $175^\circ\text{C}$ , Yield: 0.608 g, (62%).

#### Preparation of $[(\text{C}_6\text{H}_5)_4\text{As}][(\text{C}_6\text{F}_5)_2\text{SbCl}_3\text{NCS}]$ (9)

$(\text{C}_6\text{H}_5)_4\text{AsNCS}$  (4.41g; 0.01 mol) and  $(\text{C}_6\text{F}_5)_2\text{SbCl}_3$  (5.62 g; 0.01 mol) were refluxed in methanol (60 ml) in 1:1 molar ratio for 3 hrs. The resulting solution was filtered, concentrated and cooled over night to afford a crystalline compound. It was recrystallized from methanol and diethyl ether mixture (1:1). M.P.:  $150^\circ\text{C}$ , Yield: 0.652 g, (65%).

Table 1: Preparation and properties of Bis(Pentafluorophenyl)Antimony(v) anionic complexes

S. No.	Complex	Ligand	Recrystallisation solvent	Molar ratio	M.P. (°C)	Colour	Yield	
							(g)	(%)
1.	$[(C_4H_9)_4][(C_6F_5)_2SbCl_3I]$	$(C_4H_9)_4NI$	Petroleum ether (40-60°C)	1:1	170	Light yellow	0.745	80
2.	$[(CH_3)_4N][(C_6F_5)_2SbCl_3I]$	$(CH_3)_4NI$	Benzene	1:1	160	White	0.526	69
3.	$[(CH_3)_4N][(C_6F_5)_2SbCl_3Cl]$	$(CH_3)_4Cl$	Methanol	1:1	140	Off white	0.477	71
4.	$[(C_6H_5)_4Sb][(C_6F_5)_2SbCl_3Br]$	$(C_6H_5)_4SbBr$	Chloroform	1:1	125	White	0.750	70
5.	$[(C_6H_5)_4Sb][(C_6F_5)_2SbCl_3NCS]$	$(C_6H_5)_4SbNCS$	Methanol+ether	1:1	165	White	0.787	75
6.	$[(C_6H_5)_4Sb][(C_6F_5)_2SbCl_3N_3]$	$(C_6H_5)_4SbN_3$	Methanol+ether	1:1	180	Brown	0.744	72
7.	$[(C_6H_5)_4As][(C_6F_5)_2SbCl_3Br]$	$(C_6H_5)_4AsBr$	Benzene	1:1	160	Brown	0.676	66
8.	$[(C_6H_5)_4P][(C_6F_5)_2SbCl_3Br]$	$(C_6H_5)_4PBr$	Methanol	1:1	175	Brown	0.608	62
9.	$[(C_6H_5)_4As][(C_6F_5)_2SbCl_3NCS]$	$(C_6H_5)_4AsNCS$	Methanol+ether	1:1	150	White	0.652	65
10.	$[(C_6H_5)_4As][(C_6F_5)_2SbCl_3N_3]$	$(C_6H_5)_4AsN_3$	Methanol+ether	1:1	155	White	0.691	70
11.	$[(C_6H_5)_4Sb][(C_6F_5)_2SbCl_3SeCN]$	$(C_6H_5)_4SbSeCN$	Methanol+ether	1:1	220	Off white	0.801	73

Table 2: elemental analysis of Bis(Pentafluorophenyl)Antimony(v) anionic complexes

S. No.	Complex	Empirical Formula	Found (Calcd.) %		
			C	H	N
1.	$[(C_4H_9)_4][(C_6F_5)_2SbCl_3I]$	$C_{28}H_{36}Cl_3F_{10}INSb$	36.00 (36.09)	3.02 (3.87)	1.00 (1.50)
2.	$[(CH_3)_4N][(C_6F_5)_2SbCl_3I]$	$C_{16}H_{12}Cl_3F_{10}INSb$	24.99 (25.16)	1.02 (1.57)	1.01 (1.83)
3.	$[(CH_3)_4N][(C_6F_5)_2SbCl_3Cl]$	$C_{16}H_{12}Cl_4F_{10}NSb$	28.02 (28.59)	1.01 (1.79)	1.99 (2.08)
4.	$[(C_6H_5)_4Sb][(C_6F_5)_2SbCl_3Br]$	$C_{36}H_{20}Cl_3F_{10}Sb_2$	39.98 (40.31)	1.21 (1.87)	-
5.	$[(C_6H_5)_4Sb][(C_6F_5)_2SbCl_3NCS]$	$C_{37}H_{20}Cl_3F_{10}NSSb_2$	41.97 (42.29)	1.00 (1.91)	0.99 (1.33)
6.	$[(C_6H_5)_4Sb][(C_6F_5)_2SbCl_3N_3]$	$C_{36}H_{20}Cl_3F_{10}N_3Sb_2$	41.02 (41.79)	1.01 (1.93)	3.89 (4.06)
7.	$[(C_6H_5)_4As][(C_6F_5)_2SbCl_3Br]$	$C_{36}H_{20}Cl_3BrF_{10}AsSb$	41.97 (42.15)	1.03 (1.95)	-
8.	$[(C_6H_5)_4P][(C_6F_5)_2SbCl_3Br]$	$C_{36}H_{20}Cl_3BrF_{10}PSb$	43.89 (44.03)	1.99 (2.04)	-
9.	$[(C_6H_5)_4As][(C_6F_5)_2SbCl_3NCS]$	$C_{37}H_{20}Cl_3F_{10}NSAsSb$	43.93 (44.27)	1.00 (1.99)	1.00 (1.40)
10.	$[(C_6H_5)_4As][(C_6F_5)_2SbCl_3N_3]$	$C_{36}H_{20}Cl_3F_{10}N_3AsSb$	43.02 (43.77)	1.98 (2.03)	4.00 (4.26)
11.	$[(C_6H_5)_4Sb][(C_6F_5)_2SbCl_3SeCN]$	$C_{37}H_{20}Cl_3NF_{10}SeSb$	40.01 (40.48)	1.02 (1.82)	1.00 (1.28)

**Preparation of  $[(C_6H_5)_4As][(C_6F_5)_2SbCl_3N_3]$  (10)**

$(C_6H_5)_4AsN_3$  (4.25g; 0.01 mol) and  $(C_6F_5)_2SbCl_3$  (5.62 g 0.01 mol) in 1:1 molar ratio were refluxed in methanol for a period of 3 hrs. The solution was concentrated and cooled over night in a freeze to afford a solid which was crystallized with methanol and diethyl ether mixture (1:1). M.P.: 155°C, Yield: 0.691 g, (70%).

**Preparation of  $[(C_6H_5)_4Sb][(C_6F_5)_2SbCl_3SeCN]$  (11)**

$(C_6H_5)_4SbSeCN$  (5.35 g; 0.01 mol) and  $(C_6F_5)_2SbCl_3$  (5.62 g, 0.01 mol) in 1:1 molar ratio were refluxed in methanol for a period of 3 hrs. The solution was concentrated and cooled over night in a freeze to afford a solid which was crystallized with methanol and diethyl ether mixture (1:1). M.P.: 220°C, Yield: 0.801 g, (73%).

Table 3: Molar conductance and Van't Hoff factor of Bis(Pentafluorophenyl)Antimony(v) anionic complexes

S. No.	Complex	Molar conductance ( $\text{Ohm}^{-1} \text{cm}^2 \text{mol}^{-1}$ )		Van't Hoff Factor(i)
		Acetonitrile	Methanol	
1.	$[(\text{C}_6\text{F}_5)_2\text{SbCl}_3\text{I}]$	120.4	110	1.80
2.	$[(\text{C}_6\text{F}_5)_2\text{SbCl}_3\text{I}]$	126.4	108	1.92
3.	$[(\text{C}_6\text{F}_5)_2\text{SbCl}_3\text{Cl}]$	122.4	112	2.30
4.	$[(\text{C}_6\text{F}_5)_2\text{SbCl}_3\text{Br}]$	128.6	106	1.70
5.	$[(\text{C}_6\text{F}_5)_2\text{SbCl}_3\text{NCS}]$	132.2	108	1.95
6.	$[(\text{C}_6\text{F}_5)_2\text{SbCl}_3\text{N}_3]$	132.2	114	1.76
7.	$[(\text{C}_6\text{F}_5)_2\text{SbCl}_3\text{Br}]$	128.4	117	1.86
8.	$[(\text{C}_6\text{F}_5)_2\text{SbCl}_3\text{Br}]$	126.6	123	2.24
9.	$[(\text{C}_6\text{F}_5)_2\text{SbCl}_3\text{NCS}]$	128.8	124	1.96
10.	$[(\text{C}_6\text{F}_5)_2\text{SbCl}_3\text{N}_3]$	128.8	116	2.10
11.	$[(\text{C}_6\text{F}_5)_2\text{SbCl}_3\text{SeCN}]$	126.4	112	2.12

Table 4: IR Data of Bis (Pentafluorophenyl Antimony(v) anionic complexes in ( $\text{cm}^{-1}$ )

C. No.	$\nu_{\text{asy}}(\text{Ps})$	$\nu_{\text{sym}}(\text{Ps})$	$\delta(\text{Ps})$
3	2068s	1280	670
5	2020s	865w	470w
6	2085s	1270w	648w
9	2010s	860w	462w
10	2075s	1264	655w
11	2065s	-	630w

s = strong; m = medium; w = weak

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