



Preparation, Characterisation and Reactions of Symmetrical and Unsymmetrical Tris(Pentafluorophenyl) Antimony(V) Amides

By Ram Nath Prasad Yadav

Tribhuvan University, Nepal

Abstract- Several new symmetrical and unsymmetrical amides of the types $(R_f)_3Sb(NR_2)_2$ and $(R_f)_3RSb(NR_2)$ (where, $R_f = C_6F_5$; $R = C_6H_5$ and $NR_2 =$ $\overline{NCOCH_2CH_2CO}$, $\overline{NCOC_6H_4CO}$, $\overline{NCOCOC_6H_4}$, $\overline{NC_6H_4C_6H_4}$, $\overline{N N N C_6H_4}$, $\overline{NC(S)SCH}$, $\overline{NC(CH_3Cl)NC_6H_4}$, $\overline{NC(CH_3)NCHCH}$

have been prepared by the metathesis of $(R_f)_3SbCl_2$ or $(R_f)_2RSbCl$ with the appropriate metal salts of the organic ligands. The amide derivatives were also obtained by the interaction of pentafluorophenylantimony(V) chloride with organic ligand(1:1 & 1:2 molar ratio) using triethylamine as hydrogen chloride acceptor. The newly synthesized compounds have been characterized by conventional methods. A tentative trigonal bipyramidal structure is suggested for these compounds. The failure of reaction between $(R_f)_3Sb(NR_2)_2$ and CS_2 even after prolonged refluxing may be attributed to the weak dipole nature of the CS_2 and the presence of electron withdrawing group on antimony amide.

Keywords: *tris(pentafluorophenyl)antimony(v), symmetrical, unsymmetrical, metathesis, triethylamine.*

GJSFR-B Classification : FOR Code: 030299



PREPARATION CHARACTERISATION AND REACTIONS OF SYMMETRICAL AND UNSYMMETRICAL TRIS(PENTAFLUOROPHENYL)ANTIMONY(V)AMIDES

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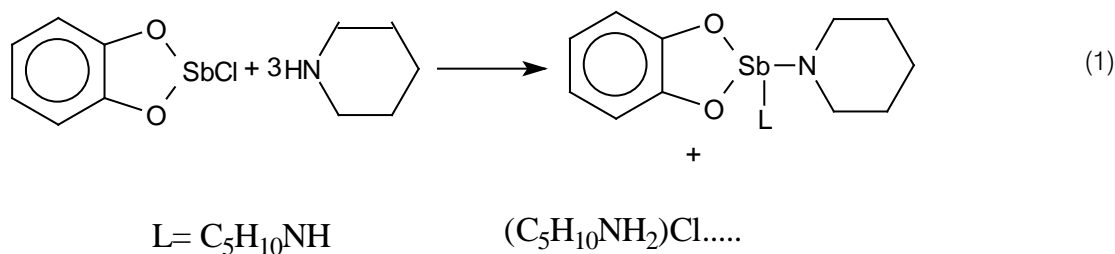
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have been prepared by the metathesis of $(R_f)_3SbCl_2$ or $(R_f)_2RSbCl$ with the appropriate metal salts of the organic ligands. The amide derivatives were also obtained by the interaction of pentafluorophenylantimony(V) chloride with organic ligand(1:1 & 1:2 molar ratio) using triethylamine as hydrogen chloride acceptor. The newly synthesized compounds have been characterized by conventional methods. A tentative trigonal bipyramidal structure is suggested for these compounds. The failure of reaction between $(R_f)_3Sb(NR_2)_2$ and CS_2 even after prolonged refluxing may be attributed to the weak dipole nature of the CS_2 and the presence of electron with drawing group on antimony amide.

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I. INTRODUCTION

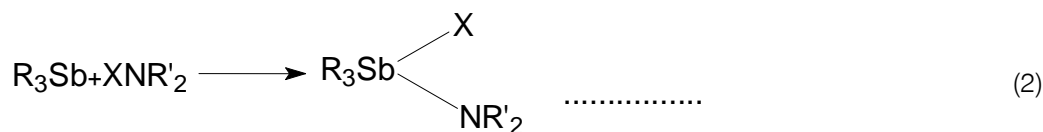
The literature reveals that the synthesis of reported amido derivatives of antimony could be accomplished via different routes. Reaction of a halogen derivatives with an amine is a common synthetic route employed for the preparation of organoantimony(V) amides derivatives are also available by this method.

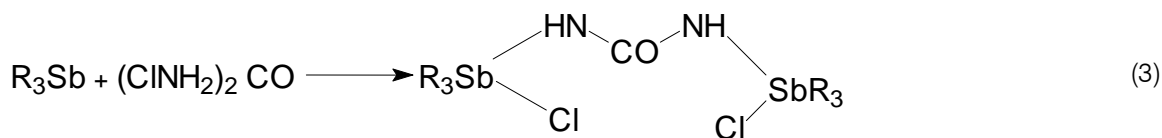


The interaction of triarylantimony dichloride and sodium amide resulted in the formation of triarylantimony(V) imides (Premraj *et al.*1984). These compounds readily give up ammonia to form compounds of higher molecular weight.

Organoantimony(V) amides $(R_3SbCl_2)NH$ ($R=Me, Et, Pr, Bu$) (Mckenny & Sisler 1967)

$R_3Sb-X\overline{NCOY}CO$ ($R=Me, Ph$; $X=Cl, Br$; $Y = (CH_2)_2, C_6H_4$ (Dahlmann & Winsel 1979) and $(Ph_3SbClNH)_2CO$ have been prepared by the oxidation of $R_3Sb(III)$ with chloroamine, haloamines or N, N-dichloro urea (eqs. 2&3), respectively.





The reaction of triphenylantimony dibromide with silver succinimide proceeded with the formation of triphenylantimony disuccinimide. Both the halogen atoms are readily replaced by two moles of succinide (Bajpai & Srivastava 1979 and Dahlmann & Winsel 1979). The synthesis and biological evaluation of some substituted tertiary arylantimony(V) diamides has been reported by Kiran Singhal *et al.* (Singhal *et al.* 1987).

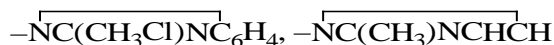
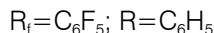
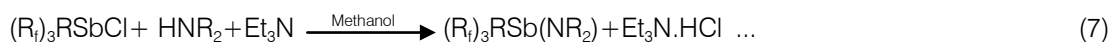
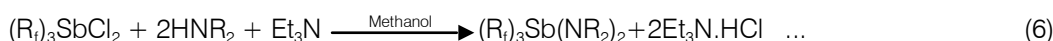
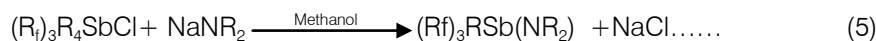
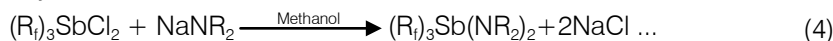
The paucity of data on the synthesis and characterization of tris(pentafluorophenyl)antimony(V) diamides and unsymmetric tetraorganostibonium amides, coupled with our interest in the synthesis and reactions of pentafluorophenylantimony(III and V) derivatives (Yadav 2012^a, Yadav 2013^a, Yadav 2013^c and Yadav 2014) and significant antimicrobial and antitumour activity of pentafluorophenylantimony(V) amides (Yadav 2012^b and Yadav 2013^b). With this in mind, we have synthesized a number of symmetrical

and unsymmetrical amides of pentafluorophenylantimony(V). The results of these studies are reported in this paper.

II. RESULT AND DISCUSSION

Tris(pentafluorophenyl)antimony(V) diamides and tris(pentafluorophenyl)phenylantimony(V) amides have been prepared by the following two methods.

1. The reaction between pentafluorophenylantimony(V) chlorides and the sodium or silver salt of the corresponding ligands in methanol solvent [eqs. (4) and (5)].
2. The interaction between pentafluorophenylantimony(V) chloride and organic ligand in the mole ratio 1:1 and 1:2 using triethylamine as hydrogen chloride acceptor in methanol solvent [eqs. (6) and (7)].

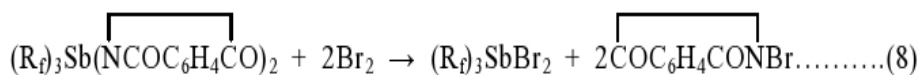


Reactions in both the cases are quite smooth and do not pose any difficulty during work-up. Amido complexes were obtained as dirty white solids and could be crystallized with organic solvent. The melting points of the newly synthesized compounds are sharp. The presence of pentafluorophenyl group imparts not only hydrolytic stability but they could be stored at room temperature, for several weeks. The consistency in melting point of representative compounds after 2 or 3

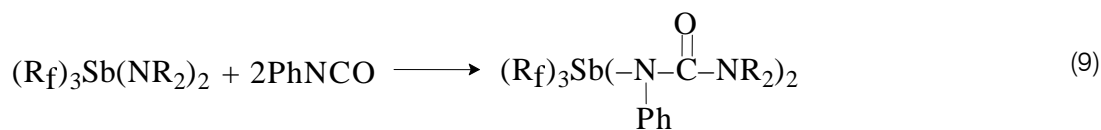
crystallization rules out the possibility of any sort of contamination or side products.

III. CLEAVAGE REACTIONS

The Sb-N bond in the newly synthesized tris (pentafluorophenyl)antimony(V) diamides, $(R_f)_3Sb(NR_2)_2$ is cleaved in preference to Sb-R_f bond by Br₂ to give corresponding halide.



Sb(NMe₂)₅ is known to react with CS₂ to give Sb(SCSNMe₂)₅. However, $(R_f)_3Sb(NR_2)_2$ failed to react with an excess of CS₂ even after prolonged refluxing on the other hand PhNCO added to the Sb-N bond in the sense of equation shown below.



The formation of the PhNCO adduct was confirmed by the absence of NCO at 2200 cm^{-1} and the appearance of a new medium intensity band at 1700 cm^{-1} due to $\nu(C=O)$. A medium band 1600 cm^{-1} may be assigned to $\nu(C-N)$. The failure of reaction of CS_2 with $(R_f)_3Sb(NR_2)_2$ may partly be attributed to the weak dipole nature of the former coupled with the presence of electron withdrawing group bonded to antimony amide. Similar course of reactions of CS_2 , RNCS and RNCO with organometalnitrogen bonded compounds has previously been reported from this laboratory by Premraj *et al.*

IV. INFRARED SPECTRA

Infrared spectra of pentafluorophenylantimony(V) compounds, $(R_f)_3SbCl_2$ and $(R_f)_3RSbCl$ has been reported earlier from the laboratory (Premraj *et al.* 1985 & Premraj *et al.* 1989). Absorption associated with various mode of vibration of C_6F_5 group corresponds well with earlier reported values and these do not vary significantly and hence not discussed here.

As has been reported earlier antimony carbon bond corresponding to Y mode for a number of pentafluorophenyl antimony compounds appears in the range of $420-480\text{ cm}^{-1}$. In the present work the appearance of a band around $460 \pm 5\text{ cm}^{-1}$ can be ascribed to antimony-carbon bond (Nunn *et al.* 1996 and Premraj & Mishra 1991).

Assignments of antimony-nitrogen frequencies are quite uncertain due to complicated nature of spectra. An antimony-nitrogen absorption band has earlier been assigned in the range $490-550\text{ cm}^{-1}$. By analogy, band appearing in the range $500-550\text{ cm}^{-1}$ are tentatively be assigned to terminal $\nu(Sb-N)$ vibrations. The comparatively weaker intensity bands located in the region $350-380\text{ cm}^{-1}$ may be due to bridging $\nu(Sb-N)$ stretching frequency (Premraj & Mishra 1991).

V. NMR SPECTRA

The 1H NMR spectra of compound (1) $(R_f)_3Sb(NCOCH_2CH_2CO)_2$ was recorded in $CDCl_3$ using TMS as the reference at 400 MHz instrument. The spectra showed a singlet at $\delta 2.60\text{ ppm}$ (due to $-CH_2CH_2-$ group) at room temperature suggesting equivalence of both the group.

^{19}F NMR of compound (1) was recorded in $CDCl_3$ taking trifluoroacetic acid as internal reference. Signal due to $F_{2,6}$ proton appeared at $\delta -130.40\text{ ppm}$ while signals due to $F_{3,5}$ and F_4 appeared at $\delta -154.78\text{ ppm}$ and $\delta -146\text{ ppm}$ respectively.

Thus on the basis of IR, 1H NMR and ^{19}F NMR spectra coupled with molecular weight, molar conductance value the newly prepared compounds can be assigned a trigonalbipyramidal structure with three C_6F_5 groups at equatorial positions and amido groups occupying axial positions.

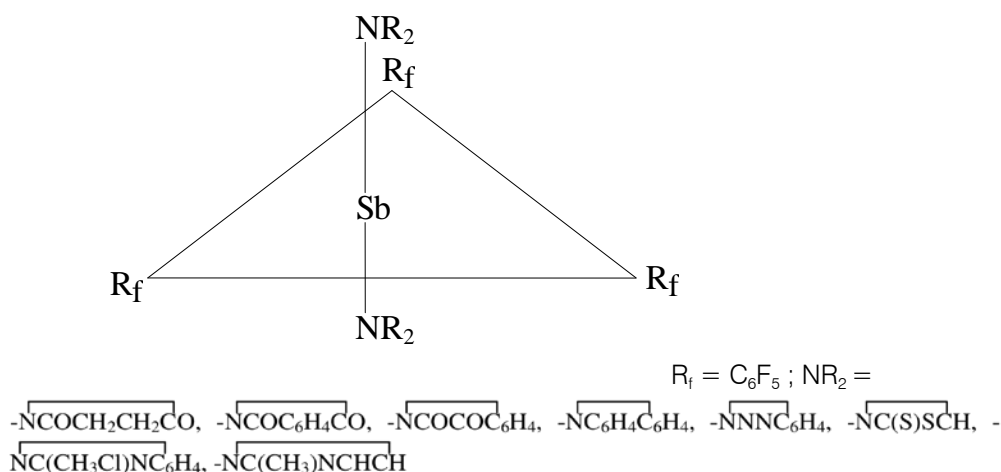


Figure 1 : Suggested structure of $(R_f)_3Sb(NR_2)_2$

VI. EXPERIMENTAL

All solvents were purified, dried and distilled before use as per the literature methods (Vogel 1989). The amides/imide moieties were procured commercially or prepared by the standard techniques and were purified by crystallization. Sodium or silver salts of the organic moieties were freshly synthesized and dried in *vacuo* before use. Tris(pentafluorophenyl)antimony(V) dichloride and tris(pentafluorophenyl)phenylantimony(V) chloride were prepared using reported methods (Premraj *et al.* 1985).

IR spectra were recorded in the range 4000-200 cm^{-1} using KBr/CsI pellets on a Perkin-Elmer 577 spectrophotometer. ^1H NMR spectra were recorded on an EM 360 L Varian spectrometer in CDCl_3 containing TMS as an external standard at room temperature. The molar conductance of 10^{-3} M solution was determined at 25°C with a PR-9500 Phillips conductivity assembly. Molecular weights were determined cryoscopically in benzene using a Beckmann thermometer of $\pm 0.01^\circ\text{C}$ accuracy. All reactions were carried out under inert and dry atmosphere. Typical synthetic procedures are given below. Relevant IR assignments, analytical data and molar conductance values are listed in Table 1-4.

a) Reactions of Tris (pentafluorophenyl) antimony(V) Chloride with Succinimide (1)

In an oxygen free atmosphere, a solution of tris(pentafluorophenyl)antimony(V) chloride (0.694g, 1 mmol) in benzene (30 ml) and succinimide (0.198g, 2 mmol) in the same solvent (30 ml) were stirred together in the presence of triethylamine at room temperature for 2h. The reactants were stirred for 1 h more to ensure completion of the reaction. $\text{Et}_3\text{N}\cdot\text{HCl}$ (m.p. 248°C) thus formed was filtered off. The filtrate on concentration in *vacuo* yielded a pale yellow crystalline solid and was recrystallized from petroleum ether ($40-60^\circ\text{C}$) to afford tris(pentafluorophenyl)antimony(V) succinimide. M.P.: 145°C ; Yield: 0.524 g (64%).

b) Reaction of Tris (pentafluorophenyl) antimony (V) Chloride with Sodium salt of 2- chloromethyl Benzoimidazole (8)

A solution of tris(pentafluorophenyl)antimony(V) chloride (0.694g, 1 mmol) in benzene (30 ml) and sodium salt of 2-chloromethylbenzoimidazole (0.377 g, 1mmol) in the same solvent were stirred together at room temperature for 3 h. NaCl thus formed was filtered off. The filtrate on concentration in *vacuo* afforded a off white crystalline solid and was recrystallized from petroleum ether ($40-60^\circ\text{C}$) to give tris(pentafluorophenyl)antimony(V) 2-chloromethyl benzoimidazole. M.P.: 145°C ; Yield: 0.541g, (69%).

c) Reaction of Tris (pentafluorophenyl) phenylantimony (V) Chloride with Carbazole (10)

In an oxygen free atmosphere, a solution of tris(pentafluorophenyl)phenylantimony(V) chloride (0.735g, 1 mmol) in benzene (30 ml) and carbazole (0.167, 1 mmol) in the same solvent (30 ml) were stirred together in the presence of triethylamine at room temperature for 2 h. The reactants were stirred for 1 h more to ensure the completion of the reaction. $\text{Et}_3\text{N}\cdot\text{HCl}$ (m.p. 248°C) thus formed was filtered off. The filtrate on concentration in *vacuo* yielded a white crystalline solid and was recrystallized from petroleum ether ($40-60^\circ\text{C}$) to afford tris (pentafluorophenyl) phenylantimony (V) carbazole. M.P.: 170°C ; Yield: 0.589 g, (68%).

d) Reaction of Tris (pentafluorophenyl) antimony (V) carbazole with carbon disulphide.

Tris(pentafluorophenyl)antimony(V) carbazole (1.906 g, 2 mmol) was refluxed with an excess of CS_2 (2 mmol) for 3 h under nitrogen atmosphere. After removal of volatiles in *vacuo* residue showed no change in the m.pt. or IR spectra as compared to the parent compound.

e) Reaction of Tris (pentafluorophenyl) antimony (V) succinimide with methanol.

Tris(pentafluorophenyl)antimony(V) succinimide was stirred with excess of methanol at room temperature (for 1 h) and then at reflux temperature (for 1 h). After removal of the volatiles the residue showed no change in m.pt. and IR spectra.

f) Reaction of Tris (pentafluorophenyl) antimony(V) isatin with water.

A mixture containing tris (pentafluorophenyl) antimony(V) isatin (1 mmol) and excess of water were refluxed for 3 h. After removal of the solvent, the residue showed no change in the m.pt. and IR spectra.

g) Reaction of Tris (pentafluorophenyl) antimony (V) phthalimide with bromine.

To a solution of Tris (pentafluorophenyl) antimony(V) phthalimide (1 mmol) in CHCl_3 (20 ml), bromine (2 mmol) in the same solvent (30 ml) was slowly added at 0°C during 30 minutes. The colour of bromine disappeared immediately after each addition. The mixture was allowed to come at room temperature and then freed from volatiles under *vacuo*. The IR spectra of the residue and TLC in benzene showed the presence of both tris(pentafluorophenyl)antimony(V) bromide and bromophthalimide. No attempts were under made to separate the products.

h) Reaction of Tris (pentafluorophenyl) phenylantimony (V) 2-methylimidazole with phenyl-isocyanate.

A mixture containing equimolar amounts of tris (pentafluorophenyl) phenylantimony(V) 2-methylimidazole (0.781 g, 1 mmol) and phenyl-

isocyanate (0.119 g, 1 mmol) were heated together for 4 h. The resulting brown liquid was treated with petroleum ether (60-80°) to afford light brown crystals of the PhNCO adduct. M.P.: 135°C; Yield: 0.584 g, (65%).

Table 1 : Preparation and Properties of Tris(Pentafluorophenyl) and Phenylantimony(V) Amides

C. No.	Complex (R _f) ₃ Sb(NR ₂) ₂	Ligand (g)	Recrystallisation solvent	Molar Ratio	M.P. (°C)	Colour	Yield (g)	Yield (%)
1.	(R _f) ₃ Sb(-NCOCH ₂ CH ₂ CO) ₂ R _f = C ₆ F ₅	HNCOCH ₂ CH ₂ CO (0.198g)	Petroleum-ether (40-60°C)	1:2	145	Pale yellow	0.524	64
2.	(R _f) ₃ Sb(-NCOC ₆ H ₄ CO) ₂	NaNCOC ₆ H ₄ CO (0.338 g)	Petroleum-ether (40-60°C)	1:2	190	Off white	0.567	62
3.	(R _f) ₃ Sb(-NCOCOC ₆ H ₄) ₂	HNCOCOC ₆ H ₄ (0.294 g)	Petroleum-ether (40-60°C)	1:2	200	Off white	0.613	67
4.	(R _f) ₃ Sb(-NC ₆ H ₄ C ₆ H ₄) ₂	AgNC ₆ H ₄ C ₆ H ₄ (0.548g)	Petroleum-ether (40-60°C)	1:2	210	Light brown	0.659	69
5.	(R _f) ₃ Sb(-N ₂ NC ₆ H ₄) ₂	HNNNC ₆ H ₄ (0.238 g)	EtOH	1:2	130	Pale yellow	0.498	58
6.	(R _f) ₃ Sb(-NC(S)SCH) ₂	HNC(S)SCH (0.334 g)	EtOH	1:2	110	Off white	0.489	59
7.	(R _f) ₃ Sb(-NC(CH ₃)NCHCH) ₂	HNC(CH ₃)NCHCH (0.164g)	CHCl ₃	1: 2	205	Off white	0.525	55
8.	(R _f) ₃ Sb(-NC(CH ₂ Cl)NC ₆ H ₄) ₂	NaNC(CH ₂ Cl)NC ₆ H ₄ (0.377g)	EtOH	1:2	145	Off white	0.541	69
9.	(R _f) ₃ Sb(-NCOCH ₂ CH ₂ CO) ₂ R _f = C ₆ F ₅ ; Ar = C ₆ H ₅	NaNCOCH ₂ CH ₂ CO (0.121g)	Petroleum-ether (40-60°C)	1: 1	150	White	0.558	70
10.	(R _f) ₃ ArSb(-NC ₆ H ₄ C ₆ H ₄) ₂	HNC ₆ H ₄ C ₆ H ₄ (0.167 g)	Petroleum-ether (40-60°C)	1:1	170	White	0.589	68
11.	(R _f) ₃ ArSb(-NC(S)SCH) ₂	AgNC(S)SCH (0.274 g)	EtOH	1:1	165	Pale yellow	0.466	58
12.	(R _f) ₃ ArSb(-NC(CH ₃)NCHCH) ₂	HNC(CH ₃)NCHCH (0.082 g)	CHCl ₃	1:1	172	Off white	0.422	54

Table 2 : Elemental Analysis of Tris(Pentafluorophenyl) and Phenylantimony(V) Amides

C. No.	Complex $(R_f)_3Sb(NR_2)_2$	Empirical Formula	Found (Calcd) %		
			C	H	N
1.	$(R_f)_3Sb(-NCOCH_2CH_2CO)_2$	$C_{26}H_8F_{15}N_2O_4Sb$	37.11 (38.11)	0.97 (0.98)	2.42 (3.42)
2.	$(R_f)_3Sb(-NCOC_6H_4CO)_2$	$C_{34}H_8F_{15}N_2O_4Sb$	42.60 (44.60)	0.86 (0.88)	2.06 (3.06)
3.	$(R_f)_3Sb(-NCOCOC_6H_4)_2$	$C_{34}H_8F_{15}N_2O_4Sb$	43.60 (44.60)	0.85 (0.88)	2.00 (3.06)
4.	$(R_f)_3Sb(-NC_6H_4C_6H_4)_2$	$C_{42}H_{16}F_{15}N_2Sb$	50.75 (52.79)	0.97 (1.68)	1.49 (2.93)
5.	$(R_f)_3Sb(-NNNC_6H_4)_2$	$C_{30}H_8F_{15}N_6Sb$	39.97 (41.92)	0.89 (0.93)	8.78 (9.78)
6.	$(R_f)_3Sb(-NC(S)SCH)_2$	$C_{22}H_2F_{15}N_2S_4Sb$	30.74 (31.86)	0.19 (0.24)	2.35 (3.38)
7.	$(R_f)_3Sb(-NC(CH_3)NCHCH)_2$	$C_{26}H_{10}F_{15}N_4Sb$	37.76 (39.76)	0.99 (1.27)	6.44 (7.14)
8.	$(R_f)_3Sb(-NC(CH_2Cl)NC_6H_4)_2$	$C_{34}H_{12}F_{15}N_4C_2Sb$	40.78 (42.78)	0.98 (1.26)	4.81 (5.87)
9.	$(R_f)_3Sb(-NCOCH_2CH_2CO)$	$C_{28}H_9F_{15}NO_2Sb$	41.12 (42.12)	1.00 (1.13)	1.25 (1.75)
$R_f = C_6F_5$; $Ar = C_6H_5$					
10.	$(R_f)_3ArSb(-NC_6H_4C_6H_4)$	$C_{36}H_{13}F_{15}NSb$	48.90 (49.90)	0.99 (1.50)	0.98 (1.62)
11.	$(R_f)_3ArSb(-NC(S)SCH)$	$C_{26}H_6F_{15}NS_2Sb$	37.87 (38.87)	0.56 (0.75)	0.74 (1.74)
12.	$(R_f)_3ArSb(-NC(CH_3)NCHCH)$	$C_{28}H_{10}F_{15}N_2Sb$	42.04 (43.04)	0.97 (1.28)	2.59 (3.59)

Table 3: IR Data of Tris(Pentafluorophenyl) and Phenyl Antimony(V) Amides (Cm⁻¹)

C. No.	$\nu(\text{Sb-C})$	$\nu(\text{Sb-N})^t$	$\nu(\text{Sb-N})^b$
1.	425	502	335
2.	435	510	340
3.	440	520	345
4.	438	515	355
5.	450	522	350
6.	460	535	365
7.	455	540	362
8.	475	545	
9.	470	550	370
10.	430	538	376
11.	428	537	373
12.	426	540	380

t = terminal; *b* = bridging

Table 4: Molar Conductance [10^{-3}m Solution] and Molecular Weight of Tris(Pentafluorophenyl) and Phenyl Antimony(V) Amides

C. No.	Molar conductance (Ohm ⁻¹ cm ² mol ⁻¹) in Acetonitrile	Molecular weight Found (Calcd.)
1.	15.21	1111.25 (818.75)
2.	17.20	1341.22 (914.75)
3.	19.23	1213.29 (914.75)
4.	20.78	1220.84 (945.75)
5.	22.79	1319.25 (858.75)
6.	27.22	1020.25 (828.75)
7.	25.49	1302.45 (953.65)
8.	26.52	1210.25 (784.75)
9.	28.29	1125.25 (797.75)
10.	30.26	1200.30 (865.75)
11.	29.27	1123.65 (802.75)
12.	27.58	1290.50 (780.75)

VII. ACKNOWLEDGEMENT

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