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By Ram Nath Prasad Yadav

Tribhuvan Uiniversity, Nepal

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Preparation, Characterisation and Reactions of Stable Adducts of Tris (Pentafluorophenyl) Antimony (V) Diisothiocyanates

Ram Nath Prasad Yadav

Abstract- A series of new stable adducts of tris (pentafluorophenyl)antimony(V) diisothiocyante of the type $(C_6H_5)_3Sb(NCS)_2.L$. Where, L = dipropyl formamide (DPF), 3-methylpyridine(3-Picoline), dimethyl formamide (DMF), tryphenylphosphine oxide(Ph_3PO), triphenylarsine oxide (Ph_3AsO), dimethyl sulfoxide (DMSO), thiourea (TU), pyridine(C_5H_5N) have been synthesized by the reaction of tris (pentafluorophenyl)antimony(V) diisothiocyanate with desired ligand in anhydrous methanol. Tris(pentafluorophenyl) antimony diisothiocyanate was obtained by the metathetical reaction of tris(pentafluorophenyl) antimony (V) dichloride and potassium thiocyanate in anhydrous methanol. The molecular weight measurement and molar conductance data of the complexes revealed them to be monomeric and non-conducting in nature. Elemental analysis and IR data indicates that the complexes have hexacoordinated octahedral structure.

Keywords: tris(pentafluorophenyl) antimony, stable adduct, diisothiocyanate, elemental analysis, IR spectra, hexacoordinate, octahedral.

I. Introduction

The Lewis acidity of pentavalent organoantimony compounds, R_nSbX_{5-n} has extensively been investigated in the last two decades by various groups of workers. The studies are not confined to R_2SbCl_3 and $RSbCl_4$ having more chlorine content but has been extended to R_3SbCl_2 derivatives as well. The latter class of compounds, based on hydrocarbon ligands are not good acceptors, but the introduction of CF_3 and C_6F_5 groups on to the metal atom (Sb) considerably enhances the Lewis acidity as evident by the formation of hexacoordinate complexes of the type $(CF_3)_3SbCl_2.L$ and $(C_6F_5)_3SbCl_2.L$. The synthesis and stereochemistry of tris(pentafluorophenyl)antimony(V) dichloride with a number of ligands viz. Dimethyl formamide, diphenyl formamide, triphenylphosphine oxide, arsine oxide, pyridine, picolines, thiourea. etc. has been reported(Singhal et al. 2002). An octahedral environment around antimony has tentatively been proposed for such complexes. On the basis of analytical, and spectroscopic data, it may be noted that except for a single reference on the formation and characterisation of $(C_6F_5)_3SbCl_2.L$, no other study related to the synthesis of molecular adducts has been reported to date (Agarwal 1990).

In view of our interest in the chemistry of pentafluorophenylantimony (III and V) derivatives (Yadav 2012[a], Yadav 2013[a], Yadav 2013[b] and Yadav 2014) and various other aspects of fluorocarbon based organoantimony compounds including their antimicrobial and antitumour activity (Yadav 2012[b] and Yadav 2013[c]), coupled with the paucity of published data in the field, we have synthesised a series of neutral adducts of tris (pentafluorophenyl) antimony (V) diisothiocynate, $(C_6F_5)_3Sb(NCS)_2$, with oxygen, nitrogen and sulphur donor Lewis bases. A few complexes of $(C_6F_5)_3SbCl_2$ have also been synthesised for the sake of comparison. The results of this investigation are reported in this paper.

II. Result and Discussion

Tris (pentafluorophenyl) antimony (v) diisothiocyanate obtained by the metathetical reaction of $(C_6F_3)_3SbCl_2$ and potassium thiocyanate, recrystallized and dried before use, was treated with the desired ligand in equivalent molar ratio in anhydrous methanol. The reactions were carried out under anhydrous oxygen free conditions.

$$(C_6F_5)_3SbCl_2 + 2KNCS \xrightarrow{\text{Methanol}} (C_6F_5)_3Sb(NCS)_2 \ldots\ldots\ldots \quad (1)$$

$$(C_6F_5)_3Sb(NCS)_2 + L \xrightarrow{\text{Methanol}} (C_6F_5)_3Sb(NCS)_2.L \ldots\ldots\ldots\ldots \quad (2)$$

Where, L = DPF, 3-Picoline, DMF, Ph_3PO, Ph_3AsO, DMSO, TU, C_5H_5N

All the reactions were found to proceed smoothly under mild conditions. The completion of the reaction takes place within 3 hrs. In most of the cases products were obtained as solid after evaporating the solvent which were crystallized with petroleum ether (40-60°C) or the mixture of diethyl ether and petroleum ether (60-80°). The complexes are soluble in common organic solvents such as chloroform, acetonitrile etc. They show monomeric constitution in freezing benzene. The complexes are

Author: Department of Chemistry, Tribhuvan University, Thakur Ram Multiple Campus, Birganj, Nepal. e-mail: ramnathy@rocketmail.com

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stable, non-susceptible to oxygen and can be stored for several weeks without decomposition. The constancy in melting point after repeated crystallization as well as TLC run in polar solvent with a single spot excluded the presence of mixture of reactants. Elemental analysis, conductance and molecular weight data are given in table (2 & 3) and correspond well to the proposed formulation of the complexes. The observed values of molecular weight indicate their monomeric constitution while the values of molar conductance of 10^{-3}M solution in acetonitrile ranges between 20-30 Ohm^{-1} cm^2 mol^{-1} at room temperature (30°C) which shows the absence of ionic species in solution.

a) Infrared Spectra

All the complexes, listed in Table 1, were characterised in the solid state by their infrared spectra in the region 4000-200 cm^{-1}. Important IR frequencies for the complexes together with their assignments are listed in Table 4. These assignments have been made by comparing the spectra in 4000-200 cm^{-1} region in the solid state of the complexes with those of the free ligands. The infrared absorptions due to pentafluorophenyl groups bonded to antimony are almost identical and do not differ significantly from those observed for other pentafluorophenyl antimony compounds reported earlier from this laboratory (Premraj et al. 1989).

b) Infrared Spectra of the Adducts with Oxygen Donors

The ν(C=O) modes in various amides bases appearing at 1650 ± 15 cm^{-1} undergo negative shift and are identified at 1608 ± 10 cm^{-1} in the spectra of the adducts suggesting weakening of the C=O bond and coordination through the oxygen atom of the base. On the basis of - Δν(CO), the DPF was found better donors as compared to DMF (Premraj & Mishra 1991).

An absorption of strong intensity for ν(S=O), ν'(AS=O) and ν(P=O) lying at 1045, 880 and 1195 cm^{-1} respectively, in the spectra of the free ligands undergoes a distinct negative shift on complexation. The corresponding absorption in the spectra of the adducts appears at 940, 835 and 1162 cm^{-1} suggesting coordination from oxygen atom of the base. The relative donor abilities of the ligand as apparent from the value of - Δν(C=O), follow the sequence DMSO > Ph_3AsO > Ph_3PO. On the basis of present and some previous studies a medium strong band in the region 380-410 cm^{-1} is assigned to ν(Sb-O) stretching frequency (Premraj & Mishra 1991).

c) Infrared Spectra of the Adducts with Nitrogen Donors

The ν(CN) frequency in $(C_6F_5)_3SbCl_2Py$ and $(C_6F_5)SbCl_2$.3-Pic is seen to decrease significantly to 1610 ± 5 cm^{-1}. In addition to this a band at 3310±10 cm^{-1} assignable to ν(NH) mode in free ligand is shifted to slightly lower frequency 3010 ± 20 cm^{-1} (Premraj & Mishra 1991). In the IR spectra of the ligand the assignment of the Sb-N bond is tentatively assigned at about 385 ± 5 cm^{-1}.

d) Infrared Spectra of the Adducts with Sulphur Donor

In sulphur donor ligand (TU) an absorption at 1069 cm^{-1} reported to posses equal contribution from ν (CN) and ν(CS). This remains unaffected on adduct formation and appears at 1075 cm^{-1}. When coordination occurs through sulphur atom, the ν(CN) suffers a positive shift while the ν(CS) suffers an almost equal negative shift. As a consequence to this the resulting absorption remains apparently unchanged (Premraj & Mishra 1991). The positive shift of ν(NH) from 3360 cm^{-1} and 3300 cm^{-1} in free ligand to 3410 and 3370 cm^{-1} in its adduct indicates absence of coordination through N-atom of the ligand and indirectly suggest Sb ← S bonding. However, on the basis of some previous observation and present studies, the (Sb-S) bond is assigned at 380 cm^{-1} (Premraj & Mishra 1991).

The diagnostic frequencies due to NCS group bound to antimony appear around at 2080, 840 and 475 cm^{-1} which could be attributed to asymmetric (NCS), symmetric (C-S) and bonding mode δ NCS, respectively. The pattern and intensity does not show any significant change reported earlier for $(C_6F_5)_3Sb(NCS)_2$ compounds (Premraj et al. 1985). Sb–C bond appears in the range 445-465 cm^{-1} (Hall & Sowerby 1988 and Nunn et al. 1996).

e) Stereochemistry of the Neutral Molecular Adducts $(Rf)_3Sb(NCS)_2.L$

It has been assumed that the addition of a Lewis base. L, to the central atom in a trigonal bipyramidal molecule takes place in a trigonal plane and steric and electrostatic factor play an important role in determining the position of entry of L. It is well established that the more electronegative group goes to the axial position and less electronegative on equatorial positions. Therefore, base L should settle in the equatorial position. It is also supported by a tentative assignment of Sb-N band at 326 cm^{-1} appearing in all the spectra and attributed to the NCS present in the axial positions.

In view of the above idea the nucleophilic attack at the position between the two fluoro groups to produce structure (Meinema & Noltes 1976) appears to be most favourable, since Rf is less electronegative than any halogen atom directly bonded to metal.

Thus analytical, conductance measurement, molecular weight determination and IR data clearly indicates that the newly synthesized complexes have hexacoordination environment around antimony with octahedral configuration as has been suggested for $R_2SbCl_3.L$ complexes (Premraj & Mishra 1991). It is generally accepted that the tris(pentafluorophenyl)-antimony(V) diisothiocyanate have a geometry of a

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trigonal bipyramidal with two halogen atoms occupying apical positions. In adduct formation as indicated, antimony atom increases its coordination number to six, for our hexacoordinate complexes. A tentative assignments of octahedral structure may be represented as below.

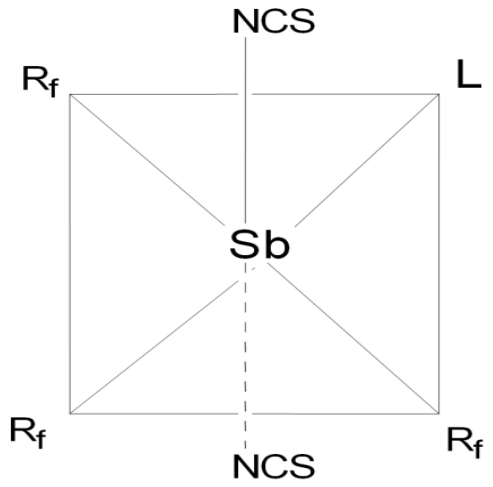

Figure : Suggested structure of $(R_f)_3Sb(NCS)_2L$

$R_f = C_6F_5$ and L = DPF, 3-Picoline, DMF, Ph_3PO, Ph_3AsO, DMSO, TU, C_5H_5N

III. Experimental

Tris (pentafluorophenyl) antimony (v) dichloride was prepared by passing chlorine gas into the solution of tris(pentafluorophenyl) antimony in pet-ether and tris (pentafluorophenyl) antimony(V) diisothiocyanate was prepared by the metathesis of tris(pentafluorophenyl)-antimony(V) dichloride and potassium thiocyanate. All the ligands were of reagent grade and used without further purification. All solvents were purified and dried by standard procedures (Vogel 1971).

The molar conductance of 10 — 3M solutions was determined at 25°C with a PR-9500 Philips conductivity assembly. Molecular weights were determined cryoscopically in benzene using a Beckmann thermometer of ±0.01°C accuracy. The stoichiometry of the compounds was established by elemental analysis. Percentage of C, H and N of the compounds was obtained on a semi-microscale (using elemental analysiser Carlo Eaba 1106, Thomas CH and Coleman analyser.

Details of the typical representative experiments are described below, the analytical data, molar conductance and IR assignments are summarised in Table 1- 4.

a) Reaction of $(C_6F_5)_3Sb(NCS)_2$ with DMF Ligand (5)

In an oxygen free atomsphere a solution of tris(pentafluorophenyl) antimony(V) diisothiocyanate (0.7388 g, 0.5 mmol) in methanol (25 ml) and DMF (0.0731 g, 0.5 mmol) in the same solvent (25 ml) were stirred together at 80°C for 3 h. After that it was filtered off. The filtrate on concentration in vacuo yielded a white crystalline solid and was recrystallised from petroleum ether (40-60°C) to afford tris(pentafluorophenyl) antimony diisothiocyanate dimethyl formamide adduct $(C_6F_5)_3Sb(NCS)_2$.DMF. M.P.: 197°C, Yield: 0.536 g, (66%).

b) Reaction of $(C_6F_5)_3Sb(NCS)_2$ with Ph_3PO Ligand (6)

A solution of tris(pentafluorophenyl)antimony(V) diisothiocyanate (0.7388 g, 0.5 mmol) in methanol (25 ml) and a solution of the same solvent (25 ml) of triphenyl phosphine oxide (0.278 g, 0.5 mmol) were stirred together at 80°C for 3 h under nitrogen. It was filtered off and the filtrate on concentration in vacuo afforded a light brown solid and was recrystallised from solvent ether to give tris(pentafluorophenyl)- antimony-diisothiocyanate triphenylphosphine oxide adduct. M.P.: 200°C Yield: 0.569g, (56%).

Table 1 : Preparation and Properties of Stable Adducts of Tris (Pentafluorophenyl)Antimony(V) Diisothiocyanates

S. No.	Complex $(R_f)_3Sb(NCS)_2$.L.	Molar Ratio	Ligand (g) Solvent (ml)	$(R_f)_3Sb(NCS)_2$(g) Solvent (ml)	M.P. (°C)	Colour	Recrystallisation solvents
	$R_f = C_6F_5$						
1.	$(R_f)_3Sb(NCS)_2$.DPF	1:1	DPF (0.129) MeOH (25)	0.7388 MeOH (25)	195	White	Petroleum ether (40-60°C)
2.	$(R_f)_3Sb(NCS)_2$.α-Pic	1:1	α-Pic (0.073) MeOH (25)	0.7388 MeOH (25)	204	Light brown	Petroleum ether (40-60°C)
3.	$(R_f)_3Sb(NCS)_2$.β-Pic	1:1	β-Pic (0.073) MeOH (25)	0.7388 MeOH (25)	170	Light brown	Petroleum ether (40-60°C)
4.	$(R_f)_3Sb(NCS)_2$.γ-Pic	1:1	γ-Pic (0.073) MeOH (25)	0.7388 MeOH (25)	188	Light brown	Petroleum ether (40-60°C)
5.	$(R_f)_3Sb(NCS)_2$.DMF	1:1	DMF (0.073) MeOH (25)	0.7388 MeOH (25)	197	White	Petroleum ether (40-60°C)
6.	$(R_f)_3Sb(NCS)_2.Ph_3PO$	1:1	Ph_3PO (0.278) MeOH (25)	0.7388 MeOH (25)	200	Light brown	Petroleum ether (40-60°C)
7.	$(R_f)_3Sb(NCS)_2.Ph_3AsO$	1:1	Ph_3AsO (0.322) MeOH (25)	0.7388 MeOH (25)	165	White	Petroleum ether (40-60°C)

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8.	$(R_f)_3Sb(NCS)_2$.DMSO	1:1	DMSO (0.078) MeOH (25)	0.7388 MeOH (25)	180	White	Petroleum ether (40-60°C)
9.	$(R_f)_3Sb(NCS)_2$.TU	1:1	TU (0.076) MeOH (25)	0.7388 MeOH (25)	183	Off white	Petroleum ether (40-60°C)
10.	$(R_f)_3Sb(NCS)_2$.Py.	1:1	Py. (0.079) Methanol (25)	0.7388 MeOH (25)	207	Light brown	Petroleum ether (40-60°C)

Table 2 : Elemental Analysis of Stable Adducts of Tris (Pentafluorophenyl) Antimony (V) Diisothiocyanates

S.No.	Complex	Empirical formula	Found (Calcd) (%)		
			C	H	N
1.	$(C_6F_5)_3Sb(NCS)_2.(C_3H_7)_2HCON$	$C_{27}H_{15}F_{15}N_3OS_2Sb$	37.34 (37.39)	1.73 (1.79)	4.84 (4.88)
2.	$(C_6F_5)_3Sb(NCS)_2.C_6H_7N$	$C_{26}H_7F_{15}N_3S_2Sb$	37.51 (37.60)	0.84 (0.90)	5.05 (5.08)
3.	$(C_6F_5)_3Sb(NCS)_2.C_6H_7N$	$C_{26}H_7F_{15}N_3S_2Sb$	37.51 (37.60)	0.84 (0.90)	5.05 (5.08)
4.	$(C_6F_5)_3Sb(NCS)_2C_6H_7N$	$C_{26}H_7F_{15}N_3S_2Sb$	37.51 (37.60)	0.84 (0.90)	5.05 (5.08)
5.	$(C_6F_5)_3Sb(NCS)_2.HCON(CH_3)_2$	$C_{23}H_7F_{15}N_3OS_2Sb$	34.00 (34.15)	0.86 (0.88)	5.17 (5.20)
6.	$(C_6F_5)_3Sb(NCS)_2.(C_6H_5)_3PO$	$C_{38}H_{15}F_{15}N_2OS_2Sb$	44.85 (44.92)	1.48 (1.52)	2.75 (2.78)
7.	$(C_6F_5)_3sSb(NCS)_2.(C_6H_5)AsO$	$C_{38}H_{15}F_{15}N_2OS_2AsSb$	42.99 (43.02)	1.41 (1.45)	2.64 (2.70)
8.	$(C_6F_5)_3Sb(NCS)_2.(CH_3)_2SO$	$C_{22}H_6F_{15}N_2OS_3Sb$	32.32 (32.37)	0.73 (0.78)	3.43 (3.46)
9.	$(C_6F_5)_3Sb(NCS)_2.NH_2CSNH_2$	$C_{21}H_4F_{15}N_4S_3Sb$	30.93 (30.98)	0.49 (0.54)	6.87 (6.89)
10.	$(C_6F_5)_3Sb$ $(NCS)_2.C_5H_5N$	$C_{25}H_5F_{15}N_3S_2Sb$	36.69 (36.72)	0.61 (0.68)	5.14 (5.22)

Table 3 : Molecular weight, conductance measurement and yield of stable adducts of Tris(pentafluorophenyl)antimony(V) diisothiocyanates

S. No.	Complex	Molar conductance (Ohm^{-1} cm^2 mol^{-1}) acetonitrile	Molecular weight in nitrobenzene Found (calcd).	Yield	
				g	%
1.	$(C_6F_5)_3Sb(NCS)_2.HCON(C_3H_7)_2$	20.6	865.70 (867.75)	0.555	64
2.	$(C_6F_5)_3Sb(NCS)_2.\alpha$-$C_6H_7N$	22.2	825.75 (831.75)	0.574	69
3.	$(C_6F_5)_3Sb(NCS)_2.\beta$-$C_6H_7N$	22.3	825.75 (831.75)	0.582	70
4.	$(C_6F_5)_3Sb(NCS)_2.\gamma$-$C_6H_7N$	22.7	825.75 (831.75)	0.590	71
5.	$(C_6F_5)_3Sb(NCS)_2.HCON(CH_3)_2$	28.9	805.10 (811.75)	0.536	66
6.	$(C_6F_5)_3Sb(NCS)_2.(C_6H_5)_3PO$	25.2	1014.75 (1016.75)	0.569	56
7.	$(C_6F_5)_3Sb(NCS)_2.(C_6H_5)_3AsO$	24.4	1069.60 (1060.67)	0.605	57
8.	$(C_6F_5)_3Sb(NCS)_2.(CH_3)_2SO$	27.6	812.75 (816.75)	0.564	69
9.	$(C_6F_5)_3Sb(NCS)_2.NH_2CSNH_2$	30.8	813.75 (814.75)	0.578	71
10.	(C_6F_5) Sb $(NCS)_2.C_5H_5N$	29.9	815 (817.75)	0.589	72

Table 4 : IR Spectra for $(R_f)_3Sb(NCS)_2L$ (Cm^{-1})

Compd. No. (Adduct)	ν(Sb-C)	ν(Sb-S)/(Sb-O)/ (Sb-N)	ν(C=N)/ (S=O)/ ν(P-O)/(N-H)/(As-O) ligand (complex)
1	458 ms	385ms	1660 (1612)
2	461 ms	382	1615
3	458 ms	384 w	1612
4	445 ms	320 w	1610
5	447 ms	395 ms	1660 (1615)
6	465 ms	405 ms	1195 (1162)
7	459 ms	390 ms	880 (835)
8	450 ms	380 ms	1045 (940)
9	455 ms	382 w	3300 (3368)
10	459 ms	381 w	1612

ms = medium strong, w = weak

IV. Acknowledgement

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