

HETEROMETALLACYCLOPHOSPHAZENE COMPLEXES CONTAINING ORGANO/ACETATOANTIMONY(III)

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Dedicated to Prof. Sushil K Pandey on his birthday

Abstract : Spirocyclic heterometallacyclopophosphazenes complexes of the type $[N(PPh_2NR')_2SbX_n]$ and $[\{N(PPh_2NR')_2\}_2SbX_n]$ where $R' = -Ph$ or $-SiMe_3$, $X = -Ph$ or $-Cl$ or $-OAc$ and $n = 1$ or 2 has been synthesized by the reaction of bis-phenylated phosphazene ligand A, $[HN(PPh_2NPh)_2]$ and bis-silylated phosphazene ligand B, $[HN(PPh_2NSiMe_3)_2]$ with $Sb(OAc)_3$, Ph_2SbCl and $PhSbCl_2$ and in the refluxing toluene under anhydrous conditions. These pale yellow complexes are characterised by the various physico-chemical techniques *viz* elemental analysis, C, H, N, molecular weight determination, spectral studies including 1H , ^{13}C and ^{31}P NMR which indicates the monomeric nature of these complexes having bidentate mode of bonding with phosphazene ligand showing tetrahedral and square pyramidal geometry around antimony atom respectively.

Index Terms - heterometallacyclopophosphazenes, organoacetato, bis-silylated phosphazene, bis-phenylated phosphazene.

I. INTRODUCTION

Phosphazene moiety $[NPCl_2]_3$ has been considered as one of the potential precursor for producing inorganic polymers, ceramics and materials for the “high-tech” purposes^{1,2}. This six member cyclotriazaphosphazenes is the most investigated phosphorous nitrogen compound, particularly pertaining to its metathesis reaction with a wide variety of nucleophiles.³⁻⁶ Cyclometallaphosphazenes chemistry is supposed to be highly active area of research because of its versatility in physical properties as well as interest in academia. These complexes found their applications in producing non-burning textile fibres, advanced elastomers, rechargeable lithium batteries beside their multi-dimensional use as bio-medical materials. In addition to this, these phosphazene complexes also found applications as potential precursor in the field of ceramics and inorganic polymers.^{7,8}

The phosphazene unit $N=PR_2$ is iso-electronic to siloxanes group ($O=SiR_2$) that has broad range of applications as fluids, greases, resins, elastomers and emulsions. In the past, cyclometallaphosphazenes containing transition and non-transition metals are building blocks in the P–N–P ring skeleton have been reported which have created lots of interest in the academia due to their versatile structural aspects as well as physical properties.⁹ This is evident from the literature that the derivatives containing Sb–O and Sb–S linkages are well known. However, scanty information is available on Sb–N linkages particularly with P–N ring system¹⁰.

Present work is motivated by the recent development of heterometallacyclopophosphazene in which one of phosphorous atom is substituted by another heteroatom. Work in our laboratories has been concerned with the discovery and development of synthesis of novel heterometallacyclopophosphazenes which are biological active compounds. We have reported previously on the synthesis of novel heterometallacyclopophosphazenes of transition metals with potential pharmaceutical activity. These compounds may be clinically active¹¹⁻¹⁶.

As part of our continuing study and synthesis of such systems here we report an efficient synthesis of heterometallacyclopophosphazenes of antimony(III) utilizing bis-phenylated and bis-silylated phosphazene ligand and $PhSbCl_2$, Ph_2SbCl and $Sb(OAc)_3$. The preparation of novel phosphorus in the main target of this synthetic programme. In spite of the fact that much literature is published on the phosphazenes but very few communications are published regarding the synthesis of organo/acetato antimony(III) heterometallacyclopophosphazenes.^{17,18}

RESEARCH METHODOLOGY

Modified Shelank techniques, N_2 atmosphere and vacuum line were used to carry out all the manipulations. All solvents were distilled from Na/K alloy and degassed three times before use. All gases were dried in pre heated oven. The bis-phenylated phosphazene and bis-silylated phosphazene ligands were prepared by literature methods. Since the compounds are air and moisture sensitive therefore utmost precautions were taken to maintain anhydrous and inert conditions during all the experimental work. Elemental analysis (C, H, N) were carried out in the microanalytical laboratory at Indian Institute of Integrative Medicines (IIIM) Jammu. Antimony was estimated gravimetrically as SbO_2 and chlorine was estimated by Volhard’s method. Molecular weight determination was carried out cyoscopically in freezing benzene. Infrared spectra (IR) were recorded in KBr mulls in the range $4000-200\text{ cm}^{-1}$ on Perkin Daltonics 377 spectrophotometer. 1H , ^{13}C , ^{31}P NMR spectra were recorded in a Bruker Daltonics DRX 300(120 MHz) spectrophotometer using TMS as the internal reference for 1H NMR and 85% H_3PO_4 as an external reference for ^{31}P NMR.

Synthesis of $[\{N(PPh_2NR')_2\}_nSb(OAc)_{3-n}]$

1.12 g (2.00mmol) of acyclic phosphazene ligand in 30 ml of toluene was added drop wise to a toluene solution (30 ml) of 0.60g (2.00 mmol) of antimony(III) acetate, $Sb(OAc)_3$. The contents of the reaction mixture were first stirred at room temperature for 2 hours and no change in colour of the reaction mixture was observed. These were then refluxed for 6 hours and the colour of the reaction mixture was changed to pale yellow. Acetic acid formed during the course of reaction was evaporated under reduced pressure which yielded pale yellow solid in almost 92% yield. A similar methodology was followed for the synthesis of complexes in 1:2 molar ratio except different refluxing time. The synthetic and analytical details of all the complexes are summarized in **Table 1**.

Synthesis of $[N(PPh_2NR')_2SbPh_2]$, where $R' = -Ph$ or $-SiMe_3$

A weighed amount of bis-silylated phosphazene ligand $[H(NPPH_2NSiMe_3)_2]$ 1.30g, 2.32 mmol was dissolved in dried toluene and then it is added drop wise to 0.72 g (2.30 mmol) diphenylantimony(III) chloride in the presence of stoichiometric amount of triethyl amine. The contents of the reaction mixture were stirred at room temperature for about 1-2 hours. With passage of time the colour of the reaction mixture changed to yellow. The precipitates of triethylamine hydrochloride Et_3NHCl were filtered off. The excess solvent was removed from the filtrate under reduced pressure which yielded pale yellow semi-solid in good yield. These complexes were further purified by extracting them from dried n-hexane. Analogous compound $[N(PPhNPh)_2SbPh_2]$ was also prepared by similar method and their synthetic and analytical data is given Table 1.

Synthesis of $[N(PPh_2NR')_2]_nSbPhCl_{2-n}]$

To a weighed amount 0.30g(1.11mmol) Ph_2SbCl in 30 ml toluene, 1.25g(2.32mmol) bis-silylated phosphazene ligand $[H(NPPH_2NSiMe_3)_2]$ in toluene was added drop wise in the presence of 0.57ml(2.32mmol) triethylamine with the help of a dropping funnel. An immediate precipitation of triethylamine hydrochloride was observed. The contents of the reaction mixture were stirred for next 2 hours and during this time the colour of reaction mixture changed to pale yellow followed by refluxing of mixture for 3 hours. Triethylamine hydrochloride formed during the course of the reaction was filtered off. The evaporation of excess of solvent under reduced pressure yielded pale yellow semi-solid in 90% yield. The same methodology was applied for the synthesis of analogue complexes and the complexes in 1: 1 molar ratio with minor difference in stirring and refluxing time. The synthetic and analytical data of these complexes are summarised in Table 1.

IV. RESULTS AND DISCUSSION

Acyclic bis-phenylated phosphazene ligand (A), $[HN(PPh_2NPh)_2]$ and bis-silylated phosphazene ligand (B), $[HN(PPh_2NSiMe_3)_2]$, are the new members of a fundamentally important class of the nitrogen containing chelating ligands and they are known to form M-N bonds with several transition as well as non-transition metals.^{19,20}

Some interesting antimony(III) phosphazene derivatives have already been synthesized by the use of antimony(III) acetate. Keeping this in view, reactions of antimony(III) acetate with bis-silylated and bis-phenylated phosphazene ligands were carried out in different stoichiometry. These reactions are quite facile and afforded the complexes correspond to $[N(PPh_2NR')_2Sb(OAc)_2]$ and $[N(PPh_2NR')_2]_nSb(OAc)_2$. These complexes are pale yellow solids and soluble in common organic solvents like benzene, acetone, chloroform, dichloromethane and insoluble in carbon tetrachloride. These complexes are highly sensitive towards atmospheric moisture and even hydrolyse by the traces of moisture. These reactions are quite facile in toluene and the by product i.e. acetic acid wise evaporated under reduced pressure along with solvent. Elemental analysis, particularly C, H, N and Sb in these complexes were in accordance with composition.

These acyclic ligands were reacted with Ph_2SbCl in 1:1 molar ratio in the presence of equimolar amount of triethylamine under anhydrous and inert conditions, which resulted in the formation of compounds $[N(PPh_2NR')_2SbPh_2]$ as pale yellow semi-solids and possess a specific odour. They are fairly soluble in most of the common organic solvent like chloroform, dichloromethane, benzene and also in coordination solvents like DMSO, DMF, etc. however, insoluble in CCl_4 . These complexes are highly moisture sensitive. However no deterioration seems to happen on keeping them under anhydrous conditions. The elemental analyses particularly of C, H, N and Sb correspond to molecular formula of these complexes.

These acyclic bis-phenylated phosphazene ligand, $[HN(PPh_2NPh)_2]$ and bis-silylated phosphazene ligand, $[HN(PPh_2NSiMe_3)_2]$ with $PhSbCl_2$ in 1:1 molar ratio and 2:1 molar ratio in toluene under anhydrous and inert conditions have also afforded the organoantimony(III) phosphazene complexes of the type $[N(PPh_2NR')_2]_nSbPhCl_{2-n}]$. These reactions appear to proceed readily as shown by the fast change in the colour of the reaction mixture simply by stirring at room temperature. The desired products were obtained in high yield after the filtration of triethylamine hydrochloride and removal of excess of solvent under reduced pressure. These organoantimony(III)cyclophosphazenes complexes are pale yellow semi-solids. These are soluble in common organic solvents and could not be distilled out even under reduced pressure and they tend to decompose. The decomposed product however could not be identified.

IR spectra

The assignment of IR spectra of these complexes has been done on the basis of earlier reports and the comparison of the data of the initial reactants. The absence of broad band for ν NH at 3350 cm^{-1} indicated the deprotonation of the ligand and subsequently the appearance of a sharp new band in the region $575\text{-}540\text{ cm}^{-1}$ has been observed, suggestive of ν Sb-N bond formation. The bands for ν C=O stretching vibrations appears in the region $1630\text{-}1600\text{ cm}^{-1}$, which is strongly indicative of the monodentate nature of the acetate moiety while ν Sb-O frequency occurs in the region $510\text{-}490\text{ cm}^{-1}$. The characteristics absorption bands for ν P-N linkages were found in $1445\text{-}1410\text{ cm}^{-1}$ region for these complexes that are in accordance with symmetric nature of ν P-N-P ring system (**compound 1-4**).

In compounds (**5-6**) the absorption band for ν P-N in the region $1230\text{-}1040\text{ cm}^{-1}$ which is in accordance with symmetric nature of ν P-N-P ring system. The absorption band for ν N-H, which was present at $3345\text{-}3350\text{ cm}^{-1}$ in the free acyclic phosphazene ligand, gets disappeared in the newly synthesised complexes. The appearance of new symmetric stretching bands in the region $560\text{-}535\text{ cm}^{-1}$ for ν Sb-N and disappearance of a broad band for ν NH at 3345 cm^{-1} is indicative of complexation. The bands of medium intensity in the region $1420\text{-}1390\text{ cm}^{-1}$ have been assigned to ν P=N.

For complexes (**7-10**) the characteristics absorption band for ν P-N in the region $1220\text{-}990\text{ cm}^{-1}$ is observed which is in accordance with the symmetric nature of the ν P-N-P ring system. The deprotonation of acyclic ligands and formation of ν Sb-N is also observed in these complexes at $570\text{-}520\text{ cm}^{-1}$. Weak to medium intensity bands were observed in the region $380\text{-}345\text{ cm}^{-1}$ and were assigned to ν Sb-Cl. The characteristics absorption for ν P=N bands were observed in the region $1450\text{-}1380\text{ cm}^{-1}$ towards near infra red region which is indicative of complexation. The relevant IR data of these complexes is summarised **table 4**

 1H NMR

The 1H NMR spectra of these derivatives reveal the deprotonation of the acyclic phosphazene ligands, which is indicated by the absence of the chemical shift for the -NH proton in the range δ 4.5-5.0 ppm. The phenyl protons for -PPh₂ and -NPh in the complexes with bis-phenylated phosphazene ligand, $[HN(PPh_2NPh)_2]$ were found as two multiplets in the region δ 6.15-8.30 ppm and phenyl protons for -PPh₂ in the complexes with bis-silylated phosphazene ligand, $[HN(PPh_2NSiMe_3)_2]$ were found as multiplet in the region δ 6.15-8.20 ppm. The chemical shift for the methyl silyl protons of trimethyl silyl group (-SiMe₃) has been observed in their characteristics region without any

appreciable change in their positions. The appearance of chemical shift for protons of $-\text{SiMe}_3$ indicates that trimethyl silyl group remain unsubstituted.

In organoacetatoantimony(III) compounds, protons of acetate moiety were found with a marginal chemical shift towards the down field (higher ppm) in the region δ 1.8-1.9 ppm. This down field shift may be probably as a consequence of complexation between ligand and Sb atom. The chemical shift for ^1H NMR spectra of these complexes are summarized in **table 2**

^{13}C NMR

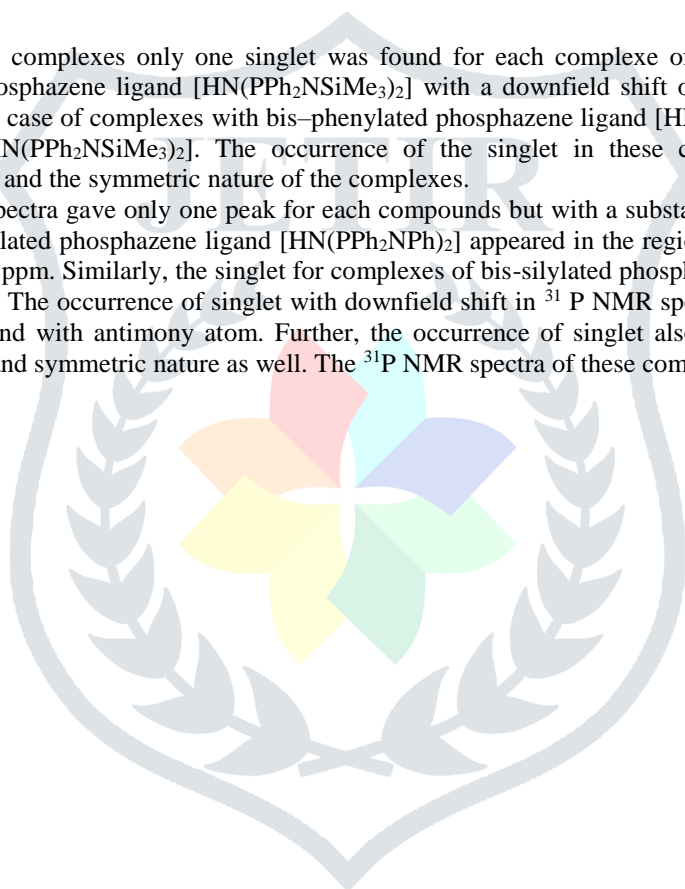
The ^{13}C NMR spectral studies at ambient temperature in CDCl_3 show only two bond coupling with phosphorous. In the spectra of these diphenylantimony(III) complexes four signals for phenyl carbons are present in the region δ 120-140 ppm attached directly to the Sb(III) atom. The appearance of singlet set for the carbons of phenyl group show equivalent nature of phenyl groups. The corrected chemical shift values (Cp-Cm, where Cp and Cm are chemical shift values of the para and meta phenyl groups) are negative in the range -4.7 to -7.7 for these complexes, this shows the electron releasing ability from metals to phenyl ring in these complexes through P-P conjugation. The carbons of other phenyl groups attached to the phosphazene ligand do not show any remarkable change from those of the parent acyclic phosphazene ligand. The carbons of trimethylsilyl (SiMe_3) group have found in their characteristics region.

^{13}C NMR spectra of organoacetatoantimony(III) complexes in CDCl_3 at ambient temperature do not differ significantly in their chemical shift values from the parent acyclic phosphazene ligands and antimony acetate. However, the ^{13}C NMR of these complexes shows the presence of carbon nuclei of acetate moiety. The appearance of the chemical shift for the acetate moiety as well as those of trimethylsilyl group indicates that complexation take place via deprotonation of the ligand instead of substitution of trimethylsilyl group. The ^{13}C NMR complexes are summarised in **table 3**.

^{31}P NMR

In the ^{31}P NMR spectra of these complexes only one singlet was found for each complex of bis-phenylated phosphazenes ligand $[\text{HN}(\text{PPh}_2\text{NPh})_2]$ and bis-silylated phosphazene ligand $[\text{HN}(\text{PPh}_2\text{NSiMe}_3)_2]$ with a downfield shift of δ 8.0 to 7.0 ppm respectively. One singlet was observed at δ 15.50 ppm in case of complexes with bis-phenylated phosphazene ligand $[\text{HN}(\text{PPh}_2\text{NPh})_2]$ and at δ 18.91 ppm with bis-silylated phosphazene ligand, $[\text{HN}(\text{PPh}_2\text{NSiMe}_3)_2]$. The occurrence of the singlet in these complexes may be correlated to the equivalence of the phosphorous nuclei and the symmetric nature of the complexes.

This proton decoupled ^{31}P NMR spectra gave only one peak for each compounds but with a substantial downfield shift in chemical shift values for the complexes of bis-phenylated phosphazene ligand $[\text{HN}(\text{PPh}_2\text{NPh})_2]$ appeared in the region δ 16-19 ppm. This value indicates a downfield shift in the range of δ 9-12 ppm. Similarly, the singlet for complexes of bis-silylated phosphazene ligand was observed in δ 15-19 ppm i.e. downfield shift of δ 4-8 ppm. The occurrence of singlet with downfield shift in ^{31}P NMR spectra of the phosphazene complexes is usually attributed to chelation of ligand with antimony atom. Further, the occurrence of singlet also explains the presence of equivalent phosphorous nucleus in the molecule and symmetric nature as well. The ^{31}P NMR spectra of these complexes are summarised **table 2**.



Figures and Tables

No suitable crystal for the purpose of single X-ray diffraction studies could be obtained. Yet on the basis of available physico-chemical studies and spectral data and in conjugation with literature^{21,22} a tetrahedral geometry in coordination number 4 for these complexes in which antimony atom is attached with bidentate ligand and two bonded groups, where as square pyramidal geometry for the complexes in Coordination number 5 may be proposed.

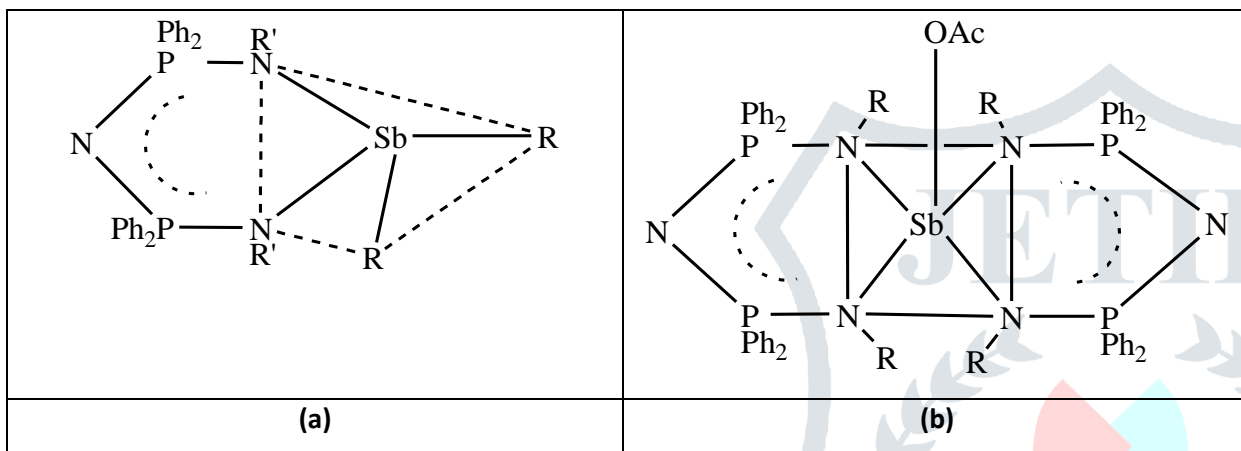


Figure 1: Proposed structures of $[N(PPh_2NR)_2SbPh_2]$, and $[N(PPh_2NR)_2SbOAc]$, where $R = -Ph$ or $-SiMe_3$

Table 1: Synthetic and Analytical data of complexes of antimony(III) cyclophosphazene

S.No	Reactants		Molar ratio (Reflux)	Product (Physical state)	Yield (%) (m.p.)	Mol.wt. Found (Cal.)	Analysis (%) Found(cal.)				
	[HN(PPh ₂ NR') ₂]	g(mmol)					Sb	C	H	N	Cl
1	1.12g (2.00 mmol)	Sb(OAc) ₃ 0.60(2.00mmol)	1:1 6 h	[N(PPh ₂ NSiMe ₃) ₂ Sb(OAc) ₂] (Pale- yellow solid)	92 (102)	790.50 (797.75)	15.2 (15.3)	51.2 (51.1)	5.5 (5.5)	5.3 (5.2)	- -
2	0.69g (1.21mmol)	Sb(OAc) ₃ 0.36(1.21mmol)	1:1 6h	[N(PPh ₂ NPh) ₂ Sb(OAc) ₂] (Pale- yellow solid)	94 (132)	815.9 (805.75)	15.1 (15.1)	59.6 (59.5)	4.4 (4.4)	5.3 (5.2)	- -
3	0.93g (1.69mmol)	Sb(OAc) ₃ 0.25(0.83mmol)	2:1 6h	[N{(PPh ₂ NSiMe ₃) ₂ }] ₂ Sb(OAc) (Pale- yellow solid)	96 (109)	1303.61 (1296.75)	9.3 (9.4)	57.3 (57.3)	6.1 (6.0)	6.4 (6.4)	- -
4	0.75g (1.32mmol)	Sb(OAc) ₃ 0.20(0.66mmol)	2:1 8h	[{N(PPh ₂ NPh) ₂ }] ₂ Sb(OAc) (Pale- yellow solid)	96 (141)	1320.50 (1312.75)	9.3 (9.2)	71.23 (71.3)	4.72 (4.7)	6.5 (6.3)	- -
5	1.30g (2.32mmol)	Ph ₂ SbCl 2.7(2.32mmol)	1:1 8h	[N(PPh ₂ NSiMe ₃) ₂ SbPh ₂] (Pale- yellow semi -solid)	89 -	825.70 (833.75)	14.5 (14.6)	- (60.4)	- (5.7)	- (5.0)	- -
6	1.41g (2.49mmol)	Ph ₂ SbCl 0.78(2.49mmol)	1:1 3h	[N(PPh ₂ NPh) ₂ SbPh ₂] (Pale- yellow semi -solid)	91 -	829.70 (841.75)	14.6 (14.4)	65.62 (65.5)	5.7 (5.7)	5.0 (4.9)	- -
7	1.31g (2.34mmol)	PhSbCl ₂ 0.63(2.34mmol)	1:1 3h	[N(PPh ₂ NSiMe ₃) ₂ SbPhCl] (Pale- yellow semi -solid)	92 -	785.75 (775.4)	15.3 (15.1)	54.4 (54.3)	6.1 (6.1)	5.3 (5.3)	4.4 (4.3)
8	1.25g (2.23 mmol)	PhSbCl ₂ 0.30(1.11mmol)	2:1 3h	[{N(PPh ₂ NSiMe ₃) ₂ }] ₂ SbPh (Pale- yellow semi-solid)	91 -	1304.51 (1314.75)	9.3 (9.2)	60.3 (60.2)	6.2 (6.3)	6.2 (6.3)	- -
9	1.10 g (1.94mmol)	PhSbCl ₂ 0.52(1.94mmol)	1:1 3h	[N(PPh ₂ NPh) ₂ SbPhCl] (Pale- yellow semi-solid)	93 -	795.50 (800.25)	15.1 (15.2)	62.9 (62.4)	4.3 (4.4)	5.2 (5.2)	4.5 (4.4)
10	0.95g (1.67mmol)	PhSbCl ₂ 0.22(0.83mmol)	2:1 4h	[{N(PPh ₂ NPh) ₂ }] ₂ SbPh (Pale- yellow semi- solid)	94 -	1312.59 (1330.75)	9.0 (9.1)	70.6 (70.3)	4.7 (4.9)	6.2 (6.3)	- -

Table 2: ¹H NMR and ³¹P NMR spectral data of antimony(III) complexes

S.No	Compound	¹ H NMR Chemical shift (in δ ppm)	³¹ P NMR Chemical shift(in δ ppm)
1	[N(PPh ₂ NSiMe ₃) ₂ Sb(OAc) ₂]	0.12,s, 18 H(-SiMe ₃) 1.85,s, 6H(-CH ₃) 6.50-8.05,m, 20H (-PPh ₂)	18.1,s
2	[N(PPh ₂ NPh) ₂ Sb(OAc) ₂]	1.95,s,6H(-CH ₃) 6.80-8.10,m,40H(-Ph)	14.1,s
3	[{N(PPh ₂ NSiMe ₃) ₂ } ₂ Sb(OAc)]	0.10,s,36H(-SiMe ₃) 1.90,s,3H(-CH ₃) 6.80-8.00,m, 40H(-Ph)	19.2,s
4	[{N(PPh ₂ NPh) ₂ } ₂ Sb(OAc)]	1.95,s,3H(-CH ₃) 6.50-8.10,m, 40H(- PPh ₂)	15.2,s
5	[N(PPh ₂ NSiMe ₃) ₂ SbPh ₂]	0.12,s,18H(-SiMe ₃) 6.20-8.10,m,20H(-PPh ₂)	18.9,s
6	[N(PPh ₂ NPh) ₂ SbPh ₂]	6.20-8.10,m,40H(-PPh ₂ and -Ph)	15.5,s
7	[N(PPh ₂ NSiMe ₃) ₂ SbPhCl]	0.12,s,18 H (-SiMe ₃) 6.20-8.20,m,25H(-PPh ₂ and -Ph)	19.1,s
8	[{N(PPh ₂ NSiMe ₃) ₂ } ₂ SbPh]	0.10,s, 36H (-SiMe ₃) 6.25-8.30,m,45H(-Ph and -PPh ₂)	18.5,s
9	[N(PPh ₂ NPh) ₂ SbPhCl]	6.15-8.25,m,35H(-Ph and -PPh ₂)	14.5,s
10	[{N(PPh ₂ NPh) ₂ } ₂ SbPh]	6.15-8.30, m, 65H (-Ph and -PPh ₂)	15.6,s

Table 3: ^{13}C NMR spectral data of antimony(III) cyclophosphazene (in δ ppm)

S. No.	Compound	-Ph	-CH ₃	C=O	C(i)	C(o)	C(m)	C(p)	C(p)-C(m)
1	[N(PPh ₂ NSiMe ₃) ₂ Sb(OAc) ₂]	120-140,m	22.5,s	75.10,s	-	-	-	-	-
2	[N(PPh ₂ NPh) ₂ Sb(OAc) ₂]	125-138,m	23.4,s	76.50,s	-	-	-	-	-
3	[{N(PPh ₂ NSiMe ₃) ₂] ₂ Sb(OAc)]	125-136,m	23.6,s	72.10,s	-	-	-	-	-
4	[{N(PPh ₂ NPh) ₂] ₂ Sb(OAc)]	120-130,m	25.7,s	75.20,s	-	-	-	-	-
5	[N(PPh ₂ NSiMe ₃) ₂ SbPh ₂]	123-139,m	-	-	139.5	134.4	132.5	125.4	-7.1
6	[N(PPh ₂ NPh) ₂ SbPh ₂]	122-140,m	-	-	138.5	132.4	130.2	124.5	-5.7
7	[N(PPh ₂ NSiMe ₃) ₂ SbPhCl]	122-140,m	-	-	140.4	137.5	132.6	126.5	-6.1
8	[{N(PPh ₂ NSiMe ₃) ₂] ₂ SbPh]	120-140,m	-	-	139.5	133.4	131.2	126.5	-4.7
9	[N(PPh ₂ NPh) ₂ SbPhCl]	125-139,m	-	-	138.4	132.5	130.6	124.3	-6.3
10	[{N(PPh ₂ NPh) ₂] ₂ SbPh]	122-139,m	-	-	140.5	134.4	133.2	125.5	-7.7

Where s= singlet, m = multiplet, i = ipso, o = ortho, m = meta and p = para

Table 4 IR spectral data of complexes of organoantimony(III) complexes(in cm^{-1})

S.No.	Compound	ν P=N	ν C=O	ν P-N-P	ν Sb-N	ν Sb-O	ν Sb-Cl
1	[N(PPh ₂ NSiMe ₃) ₂ Sb(OAc) ₂]	1420,vs	1630,vs	1220-1020	560,s	510,s	-
2	[N(PPh ₂ NPh) ₂ Sb(OAc) ₂]	1430,vs	1620,vs	1200-1015	540,s	500,s	-
3	[{N(PPh ₂ NSiMe ₃) ₂] ₂ Sb(OAc)]	1425,vs	1600,vs	1220-1015	550,s	490,s	-
4	[{N(PPh ₂ NPh) ₂] ₂ Sb(OAc)]	1445,vs	1630,vs	1210-1090	575,s	490,s	-
5	[N(PPh ₂ NSiMe ₃) ₂ SbPh ₂]	1390,vs	-	1220-1080	560,s	-	-
6	[N(PPh ₂ NPh) ₂ SbPh ₂]	1420,vs	-	1230-1040	535,s	-	-
7	[N(PPh ₂ NSiMe ₃) ₂ SbPhCl]	1410,vs	-	1200-1030	530,s	-	380,m
8	[{N(PPh ₂ NSiMe ₃) ₂] ₂ SbPh]	1405,vs	-	1210-1025	520,s	-	-
9	[N(PPh ₂ NPh) ₂ SbPhCl]	1450,vs	-	1220-1030	560,s	-	345,m
10	[{N(PPh ₂ NPh) ₂] ₂ SbPh]	1380,vs	-	1200-990	570,s	-	-

Where vs = very strong, s = strong and m = medium.

II. ACKNOWLEDGMENT

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