

**ORIGINAL RESEARCH PAPER**

**PHOSPHATO AND PHOSPHONATO ADDUCTS:  
SYNTHESIS AND SPECTROSCOPIC STUDY**

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**Abstract:** Two new adducts have been synthesized and studied by infrared and NMR spectroscopy. The suggested structures are discrete or of infinite chain type with a phosphate behaving as a bidentate ligand, a phosphonate acting as a monodentate ligand, the environments around the tin centre being tetrahedral or trigonal bipyramidal. In all the studied compounds, supramolecular architectures are obtained when hydrogen bonds are considered.

**Key words:** *discrete structures, hydrogen bonds, monodentate and bidentate, phosphate, phosphonate, supramolecular architectures, tetrahedral and trigonal bipyramidal environments*

## INTRODUCTION

The powerful coordinating ability of oxyanions is well known and has brought Hathaway to summarize the main published data on this topic [1]. Our group has yet published some papers dealing with [2-5] and initiate here the study of the interactions between ethylenediamine,  $\text{H}_3\text{PO}_4$  and  $\text{SnPh}_3\text{OH}$  or diethylenetriamine,  $\text{H}_2\text{O}_3\text{PPh}$  and  $\text{SnPh}_3\text{Cl}$  which have yielded two new adducts, infrared study of which have been carried out then structures suggested on the basis of infrared data.

## MATERIALS AND METHODS

A 1/1/1 ratio mixture of ethylenediamine (en),  $\text{H}_3\text{PO}_4$  and  $\text{SnPh}_3\text{OH}$  or a 1/1/1 ratio mixture of diethylenetriamine,  $\text{H}_2\text{O}_3\text{PPh}$  and  $\text{SnPh}_3\text{Cl}$  in ethanol are the processes to obtain (1) and (2) respectively. All the mixtures were stirred around two hours then filtered before being submitted to a slow solvent evaporation. The analytical data calculated (found) have allowed to suggest the following formulae (Table 1).

*Table 1. Suggested formulae of synthesized compounds*

Comp	Suggested formulae	Chemical composition (% mass)					
		C		H		N	
		Calc.	Found	Calc.	Found	Calc.	Found
<b>1</b>	$(\text{enH}_2)_3(\text{PO}_4)_2.\text{HPO}_4(\text{SnPh}_3)_2.6\text{H}_2\text{O}$	39.71	39.15	5.08	5.64	6.62	6.96
<b>2</b>	$\text{DETAH}_3.3\text{PhPO}_3\text{H}.\text{SnPh}_3\text{Cl}$	49.88	49.61	5.09	6.09	4.36	4.46

The elemental analyses have been obtained from the “Laboratoire de Mesures Physiques” Montpellier II University-France. The IR spectra were performed at the University of Saint Boniface-Winnipeg Canada. IR abbreviations: vs (very strong); s (strong); m (medium), w (weak).

The  $^1\text{H}$  NMR spectra were performed at the “Laboratoire de Mesures Physiques” at Montpellier II University. NMR spectra were recorded as saturated  $\text{CDCl}_3$  or DMSO at room temperature, using a Bruker 300 MHz spectrometer. The  $^1\text{H}$  NMR was measured at 300.13 MHz.  $^1\text{H}$  chemical shifts NMR are given in ppm and are referred respectively to TMS.  $^1\text{H}$  NMR abbreviations: m (multiplet), t (triplet), s (singlet). All the chemicals were purchased from QLDRIH Company-Germany and used as such.

## RESULTS AND DISCUSSION

Let us consider the:

- IR data in  $\text{cm}^{-1}$  of the two adducts:

**1:**  $\nu(\text{NH}_3)$ : 3045 (broad);  $(\nu_{\text{as}}+\nu_{\text{s}})(\text{PO}_4)$ : 1100 (vs), 1077 (vs), 1000 (vs), 970 (vs);  $(\delta_{\text{as}}+\delta_{\text{s}})(\text{PO}_4)$ : 728 (s), 696 (m), 550 (m);

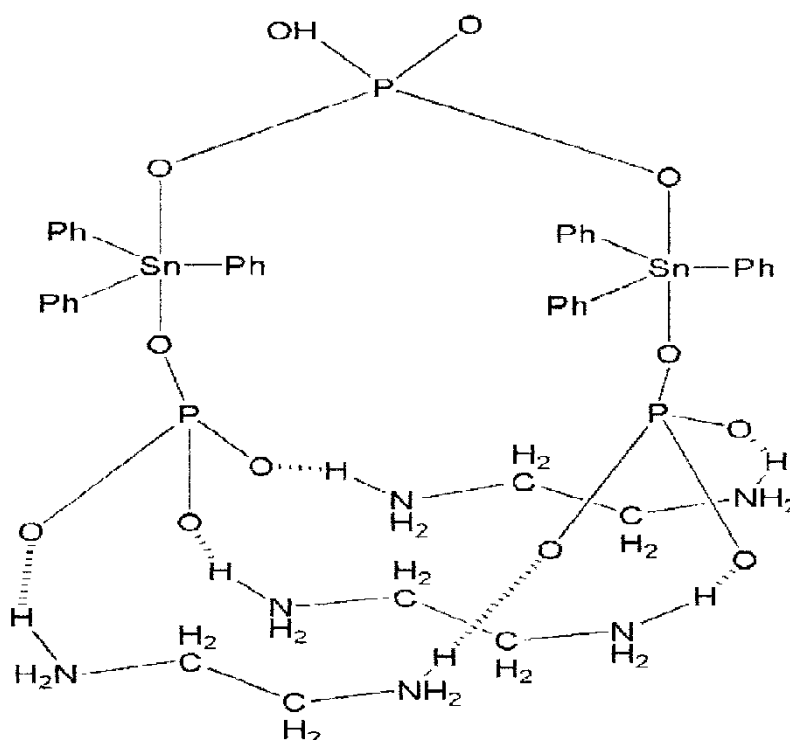
**2:**  $\nu(\text{NH}_3)$ : 3046 (broad), 2990 (broad), 2848 (broad);  $\nu(\text{PO}_3)$ : 1123 (vs), 1069 (m), 1015 (vs),  $\delta(\text{PO}_3)$ : 693 (vs).

-  $^1\text{H}$  NMR ( $\text{CDCl}_3$  or DMSO, ppm):

**1:**  $\delta$  7.08-7.82 (m, Ar-H),  $\delta$  4.26-4.28 (m,  $\text{CH}_2\text{-NH}_3$ );

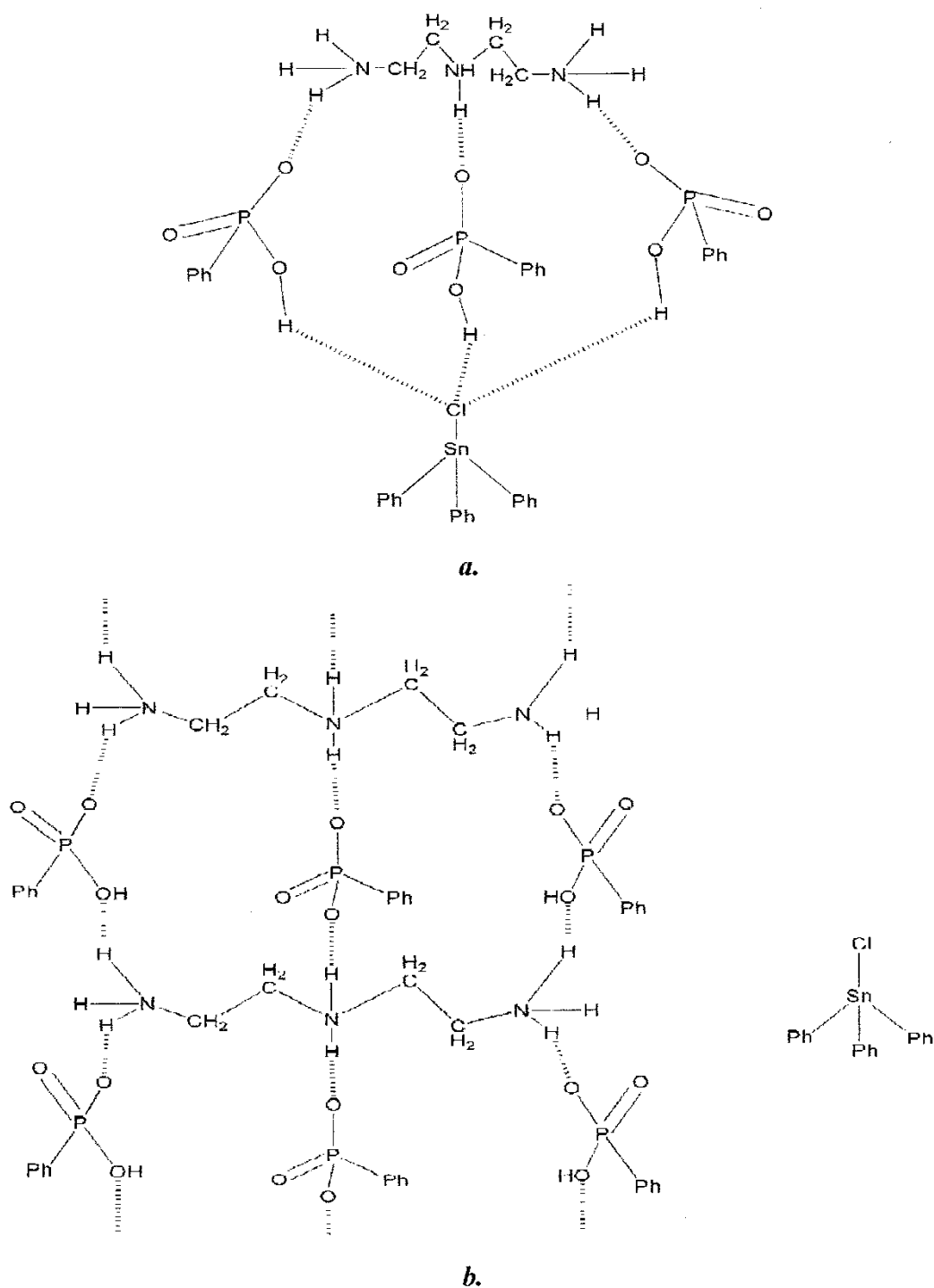
**2:**  $\delta$  7.06-7.71 (m, Ar-H).

For **1** we suggest a cyclic structure with a bidentate bridging hydrogenophosphate connecting two  $\text{SnPh}_3$  residues and monodentate phosphates linked by three  $\text{enH}_2^{2+}$ , the environment around the tin centres being trigonal bipyramidal (Figure 1).



**Figure 1.** Proposed structure for the compound **1**

For **2** the suggested structure is monomeric or of infinite chain type. In the monomeric one (Figure 2a), involved (in hydrogen bonds with the cation)  $\text{PhPO}_3\text{H}^-$  anions are linked to the  $\text{SnPh}_3\text{Cl}$  through  $\text{OH}\cdots\text{Cl}$  hydrogen bonds, the environment around the tin centre being tetrahedral. For the infinite chain (Figure 2b),  $\text{DETAH}_3\cdot 3\text{PhPO}_3\text{H}$  molecules are linked through  $\text{NH}\cdots\text{O}$  hydrogen bonds involving the cations, the  $\text{SnPh}_3\text{Cl}$  being lattice and tetrahedral.



**Figure 2.** Proposed structure for the compound 2

For all these compounds the cations can interact via hydrogen bonds leading to a supramolecular architecture.

## CONCLUSION

The studied adducts have a discrete or an infinite chain structure, the phosphate behaving as a bidentate ligand, the environment around the tin centre being tetrahedral or trigonal bipyramidal. When extra intermolecular hydrogen bonds are considered supramolecular architectures are obtained.

## ACKNOWLEDGEMENTS

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## REFERENCES

1. Hathaway, B.J.: *Comprehensive Coordination Chemistry* (Editors: Wilkinson, G., Gillard, R.D., McCleverty, J.A.), 1<sup>st</sup> ed., volume **5**, Chapter 53, Pergamon Press, Oxford, **1987**, 413;
2. Diallo, W., Diop, L., Molloy, K.C., Kociok-Köhn, G.: X-ray Structure of  $\text{HSeO}_3 \text{SnMe}_2\text{Cl}$ , *Main Group Metal Chemistry*, **2011**, **34** (3-4), 55–56;
3. Diallo, W., Okio, K.Y.A., Diop, C.A.K., Diop, L., Diop, L.A., Russo, U.: New Selenito Residues Containing Complexes and Adducts: Synthesis and Spectroscopic Studies, *Main Group Metal Chemistry*, **2009**, **32** (2), 93-99;
4. Allouch, H., Diop, L.: Synthesis and infrared study of some new  $\text{SnC}_2\text{O}_4\text{Cl}_2$  adducts and complexes *Scientific Study & Research - Chemistry & Chemical Engineering, Biotechnology, Food Industry*, **2012**, **13** (3), 317-323;
5. Gueye, N., Diop, L., Diop, L.: New  $\text{R}_2\text{NH}_2\text{OH}$  (R = Cy, Bu) adducts of  $\text{MX}_2$ ,  $\text{MX}_3$  or  $\text{MX}_5$ : Synthesis and infrared study, *Scientific Study & Research - Chemistry & Chemical Engineering, Biotechnology, Food Industry*, **2012**, **13** (4), 399–403.