

C₂O₄(SnPh₃)₂ ISOMERS AND SOME OF THEIR ADDUCTS: SYNTHESIS AND SPECTROSCOPIC CHARACTERIZATION

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Received: February, 12, 2015

Accepted: March, 30, 2015

Abstract: The study of the interactions between C₂O₄(SnPh₃)₂ and a Lewis base (Ph₃PO) or salts such as Ph₄PCl, (Bu₂NH₂)₂C₂O₄·3H₂O, (Pr₂NH₂)₂C₂O₄(Cy₂NH₂)₂C₂O₄·2H₂O have yielded seven new adducts, infrared and Mossbauer studies which have been carried out. The suggested structures are discrete or of infinite chain type. Most of the structures contain C₂O₄(SnPh₃)₂ with *cis* coordinated SnPh₃ residues characterized for the first time in this work. In Ph₃PO containing adducts the Lewis base coordinates a SnPh₃ residue. The oxalate behaves as a mono- or bidentate, a mono- or bichelating, a only hydrogen bonds involved ligand. In the structures of the compounds containing a non symmetrical cation, this one is involved in N-H...O hydrogen bonds.

Keywords: C₂O₄(SnPh₃)₂ in two different conformations, discrete and infinite chains structures; IR, Mossbauer, trans and cis coordinated SnPh₃ residues

INTRODUCTION

The synthesis of new triorganotin (IV) compounds is connected to their biological activity and industrial applications mainly [1] and explains the focus of several research groups [2 - 5]. In the framework of our seek of new organotin (IV) compounds our group has yet published several papers [6 - 9] including the X ray structure of $C_2O_4(SnPh_3)_2$ [10]. We initiate here the study of the interactions between $C_2O_4(SnPh_3)_2$ and a Lewis base (Ph_3PO) or salts such as Ph_4PCl or $(Bu_2NH_2)_2C_2O_4 \cdot 3H_2O$, $(Pr_2NH_2)_2C_2O_4$ $(Cy_2NH_2)_2C_2O_4 \cdot 2H_2O$ with $SnPh_3Cl$ which have yielded seven new adducts, infrared and Mossbauer studies of which have been carried out then structures suggested on the basis of spectroscopic data.

MATERIALS AND METHODS

The synthesis $C_2O_4(SnPh_3)_2$ had yet been reported [10]. On allowing $C_2O_4(SnPh_3)_2$ in MeOH to react with Ph_3PO or Ph_4PCl as ethanolic solutions in $\frac{1}{2}$ ratio, a solution (Ph_3PO) and a precipitate (Ph_4PCl) were obtained respectively. The solutions when submitted to a slow solvent evaporation gives a white powder [A] while the precipitate [B] stirred around two hours is filtered and collected. $(Cy_2NH_2)_2C_2O_4 \cdot 2H_2O - C = 68.99$ (69.02), H = 10.69 (10.59), N = 6.19 (6.20) - has been obtained as a powder on neutralizing completely oxalic acid with Cy_2NH in water and evaporating at 60 °C. $(Pr_2NH_2)_2C_2O_4 - C = 57.50$ (57.09), H = 11.03 (10.89), N = 9.59 (9.55) has been obtained as a precipitate on neutralizing completely oxalic acid with Pr_2NH in water. On allowing $C_2O_4(SnPh_3)_2$ to react with $(Cy_2NH_2)_2C_2O_4 \cdot 2H_2O$ both as ethanolic solutions in $\frac{1}{2}$ ratio a precipitate [C] was obtained; $(Bu_2NH_2)_2C_2O_4 \cdot 3H_2O$ has been obtained on neutralizing completely oxalic acid with Bu_2NH in water – a powder is collected after a water evaporation 60 °C. When $(Bu_2NH_2)_2C_2O_4 \cdot 3H_2O$ is mixed with $SnPh_3Cl$ in 1/1 both as ethanolic solutions, a solution is obtained; a slow solvent evaporation gives a white powder [D]. The mixture of $C_2O_4(SnPh_3)_2$ (in ethanol) with a slightly hydrated CH_3COCH_3 in 1:2 ratio gives [E]. On allowing $(Pr_2NH_2)_2C_2O_4$ to react with $SnPh_3Cl$ in $\frac{1}{2}$ ratio [F] is obtained; $(Cy_2NH_2)_2C_2O_4 \cdot 2H_2O$ when allowed to react with $C_2O_4(SnPh_3)_2$ and Ph_3PO in ethanol in 1/1/1 ratio [G] is obtained (the four last compounds are white precipitates stirred around two hours).

The analytical data calculated (found) **A** { %C = 63.03 (62.7) %H = 4.22 (4.08) %Sn = 22.13 (22.86) }; **B** { %C = 64.02 (64.05) %H = 4.30 (4.20) %Cl = 3.05 (3.02) %Sn = 20.42 (20.61) }; **C** { %C = 62.52 (62.41) %H = 7.52 (7.59) %N = 3.24 (3.26) } and **D** { %C = 58.56 (58.39) %H = 6.21 (5.15) %N = 2.48 (2.39) }. { %C = 56.62 (56.53), %H = 4.00 (3.97) } for **E**; { %C = 57.87 (57.42), H = 4.63 (4.69), N = 1.05 (1.13) } for **F** and { %C = 66.90 (66.29), H = 5.55 (5.50), N = 0.91 (0.88) } for **G** have allowed to suggest $C_2O_4(SnPh_3)_2 \cdot OPPh_3$ (**A**), $C_2O_4(SnPh_3)_2 \cdot Ph_4PCl$ (**B**), $2(Cy_2NH_2)_2C_2O_4 \cdot C_2O_4(SnPh_3)_2 \cdot 2H_2O$ (**C**), $2(Bu_2NH_2)_2C_2O_4 \cdot C_2O_4(SnPh_3)_2 \cdot 2SnPh_3Cl$ (**D**) and $C_2O_4(SnPh_3)_2 \cdot H_2O$ (**E**) $Pr_2NH_2C_2O_4SnPh_3C_2O_4(SnPh_3)_2$ (**F**) and $(Cy_2NH_2)_2C_2O_4 \cdot C_2O_4(SnPh_3)_2 \cdot 2SnPh_3OH \cdot 4Ph_3PO$ (**G**) as formulae.

The elemental analyses were performed by the *Service Central d' Analyses* - CNRS – Vernaison France, the Laboratory of Microanalyses, Department of Chemistry University of Padova – Italy, the Laboratory of Microanalyses, Department of

Chemistry University of Bath UK and the Laboratory of Microanalyses - Instituto de Quimica – UNAM - Mexico.

The infrared spectra were recorded by means of a PE 580 (4000-200 cm⁻¹) or a FTIR - Nicolet (600-50 cm⁻¹) spectrometer at the University of Padova (Italy), the sample being as Nujol mulls using CsI or polyethylene windows, Mossbauer spectra were obtained as described previously. The Mossbauer spectra were obtained as described in [11, 12]. IR abbreviations: vs (very strong), s (strong), m (medium), w (weak), vw (very weak), sh (shoulder). Mossbauer parameters are given in mm/s (abbreviations: Q.S = quadrupole splitting, I.S = isomer shift, Γ = full width at half - height, A = %). All chemicals were purchased from Aldrich and used without any further purification.

RESULTS AND DISCUSSION

Let us consider the i.r data of the studied compounds:

A: { $\nu_{as}COO^-$ (1656vs; 1671sh), ν_aCOO^- (1247vs; 1229sh), δCOO^- (773m), ($\omega+p$) COO^- (423m; 363vw), ν_sSnC_3 (218sh), νSnO (200w)};

B: { $\nu_{as}COO^-$ (1687vs; 1632vs), ν_sCOO^- (1249vs; 1236sh), δCOO^- (780m), ($\omega+p$) COO^- (481w; 361vw), ν_sSnC_3 (214w), νSnO (201w)}; νSnX (194w)};

C: { $\nu_{as}COO^-$ (1675s; 1535s), ν_sCOO^- (1295s; 1270s), δCOO^- (781m), ($\omega+p$) COO^- (485m; 449s)};

D: { $\nu_{as}COO^-$ (1687s; 1632s), ν_sCOO^- (1249s; 1236sh), δCOO^- (780m), ($\omega+p$) COO^- (484w; 447s) and the Mossbauer ones,

A - IS₁=1.41 mm·s⁻¹; QS₁=2.58 mm·s⁻¹; Γ_1 =0.92; A₁=50; IS₂=1.49 mm·s⁻¹; QS₂=3.44 mm·s⁻¹; Γ_2 =0.99; A₂=50.

B - IS₁=1.32 mm·s⁻¹; QS₁=3.25 mm·s⁻¹; Γ_1 =0.79; A₁=50; IS₂=1.10 mm·s⁻¹; QS₂=2.26 mm·s⁻¹; Γ_2 =0.79; A₂=50.

C - IS=1.05 mm·s⁻¹; QS=2.21 mm·s⁻¹; Γ =0.85; A=100;

D - IS=1.06 mm·s⁻¹; QS=2.21 mm·s⁻¹; Γ =0.88; A=100.

The Mossbauer data of [A] are consistent with the simultaneous presence of *trans* coordinated (3.44 mm·s⁻¹) and tetrahedral SnPh₃ residues (2.58 mm·s⁻¹) according to Bancroft and Platt [13] and to Parish and Platt [14]. These Mossbauer data allow to suggest in the solid state, a discrete structure with two components - C₂O₄(SnPh₃)₂ and C₂O₄(SnPh₃.L)₂ – interacting very weakly. This adduct can be considered as C₂O₄(SnPh₃)₂.2Ph₃PO adduct with one C₂O₄(SnPh₃)₂ molecule interacting with two Lewis bases, the second molecule being a lattice one as in (Figure 1).

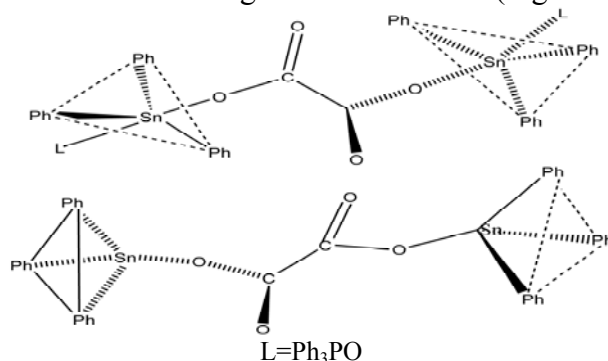


Figure 1. Proposed structure for A

NB: Compared to the succinate $(\text{SnPh}_3)_2$.dimethylformamid [15], the longer distance between the CO_2 groups minimizes the steric effects, very strong in this compound, preventing therefore strong interactions between the two components of the structure. The Mossbauer data of **[B]** are consistent with the simultaneous presence of *cis* ($2.26 \text{ mm}\cdot\text{s}^{-1}$) and *trans* ($3.25 \text{ mm}\cdot\text{s}^{-1}$) coordinated SnPh_3 residues according to Bancroft and Platt [13] and to Parish and Platt [14], allowing to suggest a structure containing $[\text{C}_2\text{O}_4(\text{SnPh}_3\text{Cl})_2]^{2-}$ (with bidentate oxalate linked to SnPh_3Cl) and $\text{C}_2\text{O}_4(\text{SnPh}_3)_2$ (containing a bichelating oxalate and *cis* coordinated SnPh_3 residues) the two entities interacting very weakly through $\text{Sn}-\text{Cl}\cdots\text{Sn}$ bonds leading to an infinite chain. $\text{C}_2\text{O}_4(\text{SnPh}_3)_2$ can also be considered as a lattice molecule interacting through Van der Waals forces with the other component of the structure (Figures 2a and 2b).

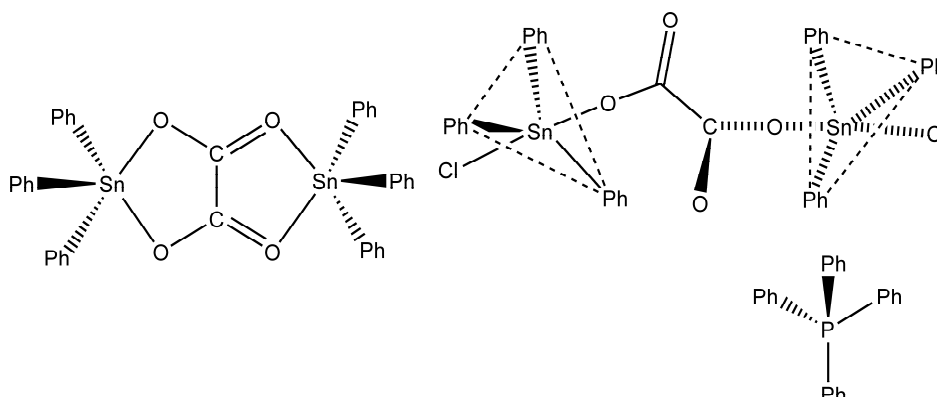


Figure 2a. Proposed structure for **B**

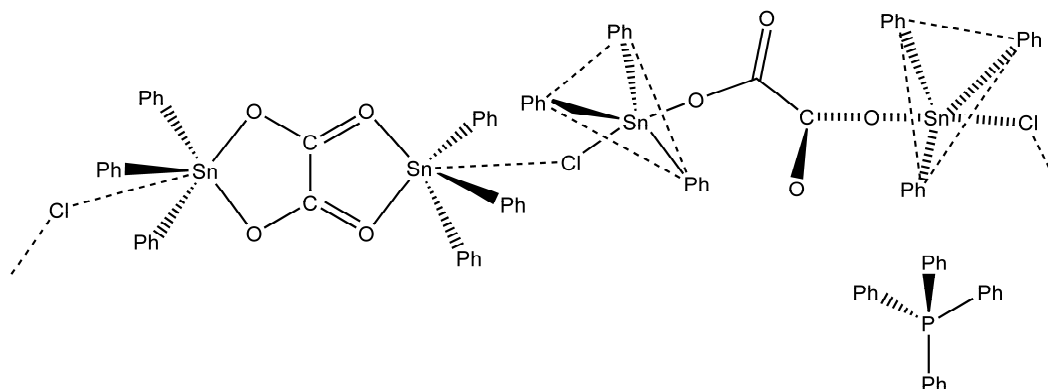


Figure 2b. Proposed structure for **B**

The dinuclear complex anion $[\text{C}_2\text{O}_4(\text{SnPh}_3\text{Cl})_2]^{2-}$ had yet been reported in $(\text{Me}_4\text{N})_2\text{C}_2\text{O}_4\cdot 2\text{SnPh}_3\text{Cl}$ [16] in which a Q.S of $2.98 \text{ mm}\cdot\text{s}^{-1}$ had been reported. In $2(\text{Cy}_2\text{NH}_2)_2\text{C}_2\text{O}_4\cdot \text{C}_2\text{O}_4(\text{SnPh}_3)_2\cdot 2\text{H}_2\text{O}$ **[C]** and $2(\text{Bu}_2\text{NH}_2)_2\text{C}_2\text{O}_4\cdot \text{C}_2\text{O}_4(\text{SnPh}_3)_2\cdot 2\text{SnPh}_3\text{Cl}$ **[D]** the value of the Q.S ($2.21 \text{ mm}\cdot\text{s}^{-1}$) in both compounds is a clear indication of the presence of $\text{C}_2\text{O}_4(\text{SnPh}_3)_2$ in a *cis* coordinated conformation and allow to suggest while involving the cations the discrete (Figure 3a) or a double infinite chain structure with $\text{C}_2\text{O}_4(\text{SnPh}_3)_2$ and the water molecules as lattice ones (Figure 3b) for **(C)**. For **(D)** the discrete structure is reported on Figure 4.

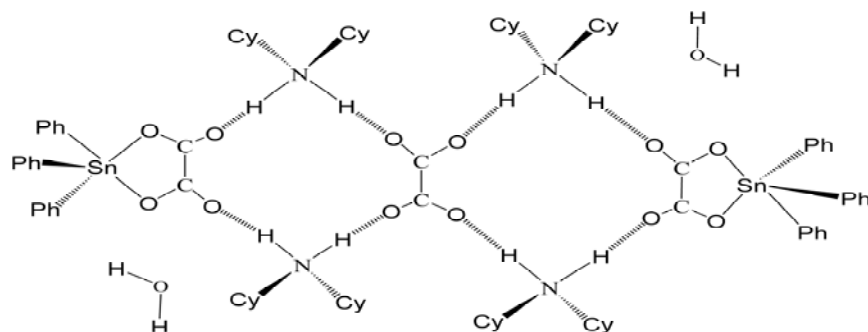


Figure 3a. Proposed structure for C

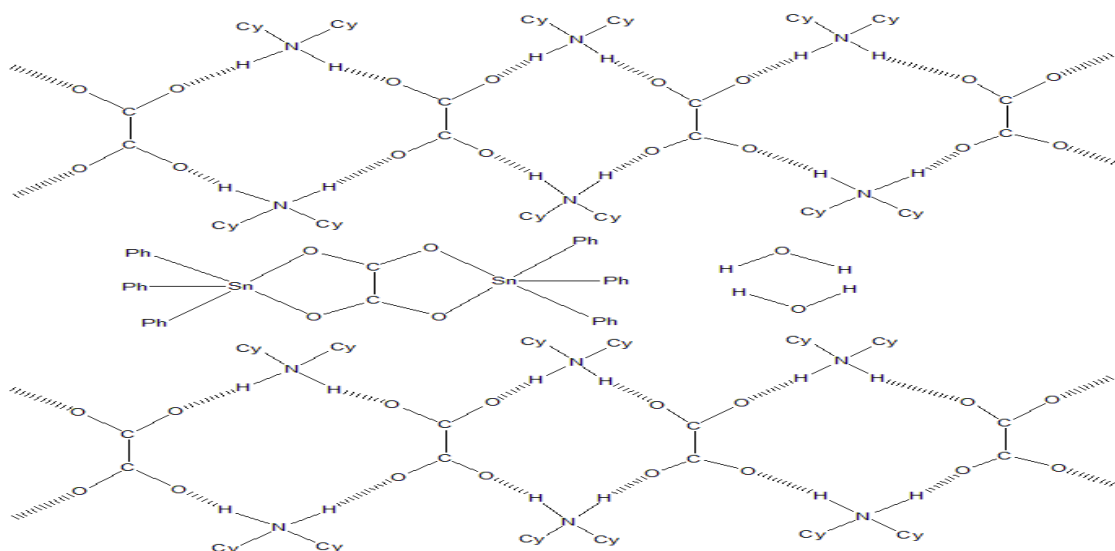


Figure 3b. Proposed structure for C

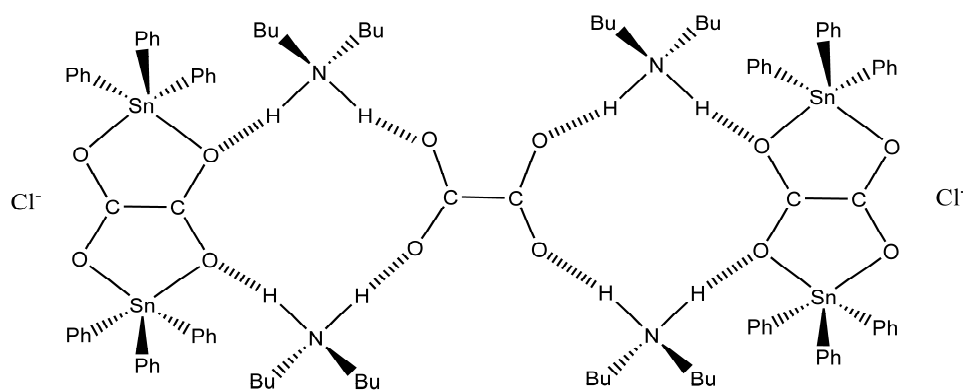


Figure 4. Proposed structure for D

Let us consider the infrared and Mossbauer data of C₂O₄(SnPh₃)₂·H₂O

E: $\nu_{\text{as}}\text{COO}^-$ (1671s, 1617m); $\nu_{\text{s}}\text{COO}^-$ (1279vs, 1239vs); δCOO^- (789s);

E - $I.S_1 = 0.87 \text{ mm}\cdot\text{s}^{-1}$; $Q.S_1 = 2.17 \text{ mm}\cdot\text{s}^{-1}$; $\Gamma_1 = 1.01$; $\%A_1 = 12$; $I.S_2 = 1.20 \text{ mm}\cdot\text{s}^{-1}$; $Q.S_2 = 2.82 \text{ mm}\cdot\text{s}^{-1}$; $\Gamma_2 = 1.01$; $\%A_2 = 88$.

From the Mossbauer data we can outline the presence of two types of C₂O₄(SnPh₃)₂, one with a Q.S of $2.82 \text{ mm}\cdot\text{s}^{-1}$ (88 %) and one with the Q.S of $2.17 \text{ mm}\cdot\text{s}^{-1}$ (12 %) these two entities being in a mixture but clearly defined. C₂O₄(SnPh₃)₂ yet reported by our

group was characterized by the presence of a doublet on its infrared spectrum around 785 due to δCOO^- while on the spectrum studied here this doublet is absent, linked to the variation of the crystal symmetry induced by the presence of the water molecule. However the value of the quadrupole splitting of $2.82 \text{ mm}\cdot\text{s}^{-1}$ very similar to the one anhydrous $\text{C}_2\text{O}_4(\text{SnPh}_3)_2$ indicates the same environment *ie* tetrahedral showing the absence of coordination by the water molecule indicating its lattice nature in the solid state. This allows to suggest for $\text{C}_2\text{O}_4(\text{SnPh}_3)_2\cdot\text{H}_2\text{O}$ a discrete structure with a bidentate oxalate, the environment around the tin centre being tetrahedral, the water molecule being lattice - this adduct is an insertion adduct (Figure 5a).

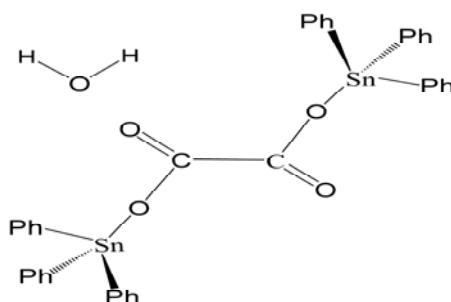


Figure 5a. Proposed structure for the main component of the compound **E**

For the minor component of **E** - $\text{C}_2\text{O}_4(\text{SnPh}_3)_2$ - ($Q.S = 2.17 \text{ mm}\cdot\text{s}^{-1}$, $A = 8 \%$) the value of the quadrupole splitting indicates *cis* coordinated of SnPh_3 residues according to [13, 14] allowing to suggest the structure reported on Figure 5b.

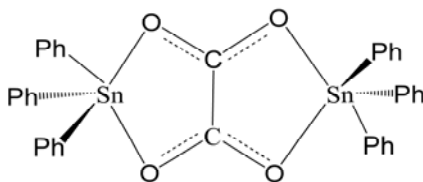


Figure 5b. Proposed structure for the minor component of the compound **E**

Let us consider the infrared and Mossbauer data of $\text{Pr}_2\text{NH}_2\text{C}_2\text{O}_4\text{SnPh}_3\cdot\text{C}_2\text{O}_4(\text{SnPh}_3)_2$

F: νasCOO^- 1672s, 1650vs; νsCOO^- : 1273vs, 1243s; δCOO^- : 784s;

F - $Q.S_1 = 2.07 \text{ mm}\cdot\text{s}^{-1}$, $IS_1 = 0.79 \text{ mm}\cdot\text{s}^{-1}$, $\Gamma_1 = 0.85 \text{ mm}\cdot\text{s}^{-1}$, $A_1(\%) = 67$; $Q.S_2 = 2.90 \text{ mm}\cdot\text{s}^{-1}$, $IS_2 = 1.07 \text{ mm}\cdot\text{s}^{-1}$, $\Gamma_2 = 0.92 \text{ mm}\cdot\text{s}^{-1}$, $A_2(\%) = 33$;

$(\text{Cy}_2\text{NH}_2)_2\text{C}_2\text{O}_4\cdot\text{C}_2\text{O}_4(\text{SnPh}_3)_2\cdot 2\text{SnPh}_3\text{OH}\cdot 4\text{Ph}_3\text{PO}$

G: νasCOO^- : 1680s, 1670s; νsCOO^- : 1308m, 1280vs, 1228s; δCOO^- : 790s;

The infrared spectra of these complexes exhibit the presence of a non-centrosymmetrical oxalate because of the presence of more than two bands in the stretching vibrations region, the wide absorption band centered in 2900 cm^{-1} indicating the presence of hydrogen bonds. On the infrared spectra of the compounds (**G**), the appearance of $\nu(\text{PO})$ as a very strong band at 1180 cm^{-1} , reveals the presence of Ph_3PO molecules.

The Mossbauer data of $\text{Pr}_2\text{NH}_2\text{C}_2\text{O}_4\text{SnPh}_3\cdot\text{C}_2\text{O}_4(\text{SnPh}_3)_2$ is consistent with the presence of a monocoordinated SnPh_3 residue ($Q.S=2.90 \text{ mm}\cdot\text{s}^{-1}$) - we remind the fact that for $\text{C}_2\text{O}_4(\text{SnPh}_3)_2$ contains monocoordinated SnPh_3 residues and has a $Q.S = 2.82 \text{ mm}\cdot\text{s}^{-1}$ [10] - and two *cis* coordinated SnPh_3 residues: this allows to suggest a two metallic components structure one of the components being an infinite chain of

Pr₂NH₂C₂O₄SnPh₃ - or an oligomer - with an oxalate monocoordinating a SnPh₃ residue and linked by NH.....O hydrogen bonds to the cation, the second component being *cis* coordinated C₂O₄(SnPh₃)₂ (Figure 6a).

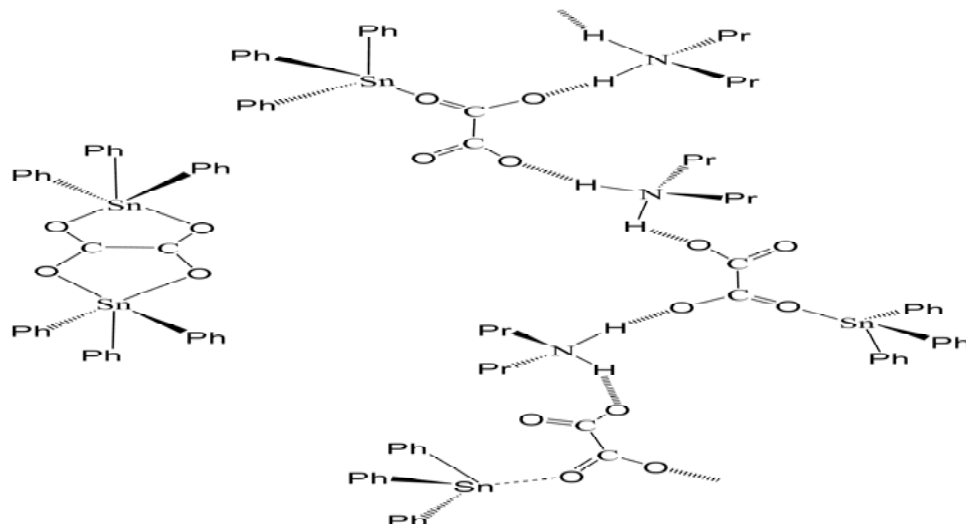


Figure 6a. Proposed structure for compound **F**

We have arbitrary chosen the case of a dimer as oligomer for the second structure while any oligomer works (Figure 6b) the two components interact through van der Waals forces.

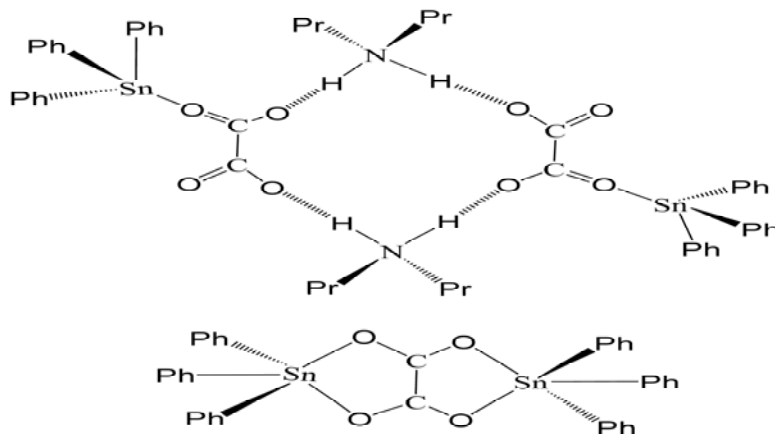


Figure 6b. Proposed structure for compound **F**

(Cy₂NH₂)₂C₂O₄·C₂O₄(SnPh₃)₂·2SnPh₃OH·4Ph₃PO can be considered as a [Cy₂NH₂C₂O₄SnPh₃·Ph₃PO]₂ hydrogen bonded dimer with trans coordinated SnPh₃ residues linked everyone to a Ph₃PO molecule; this entity is then hydrogen bonded or not to the non coordinating oxalates (Figures 7a and 7b). A third structure containing [Cy₂NH₂C₂O₄SnPh₃]₂ reported by Ng and Hook [17] interacting through Van der Waals forces with two SnPh₃OH·2Ph₃PO (Figure 7c).

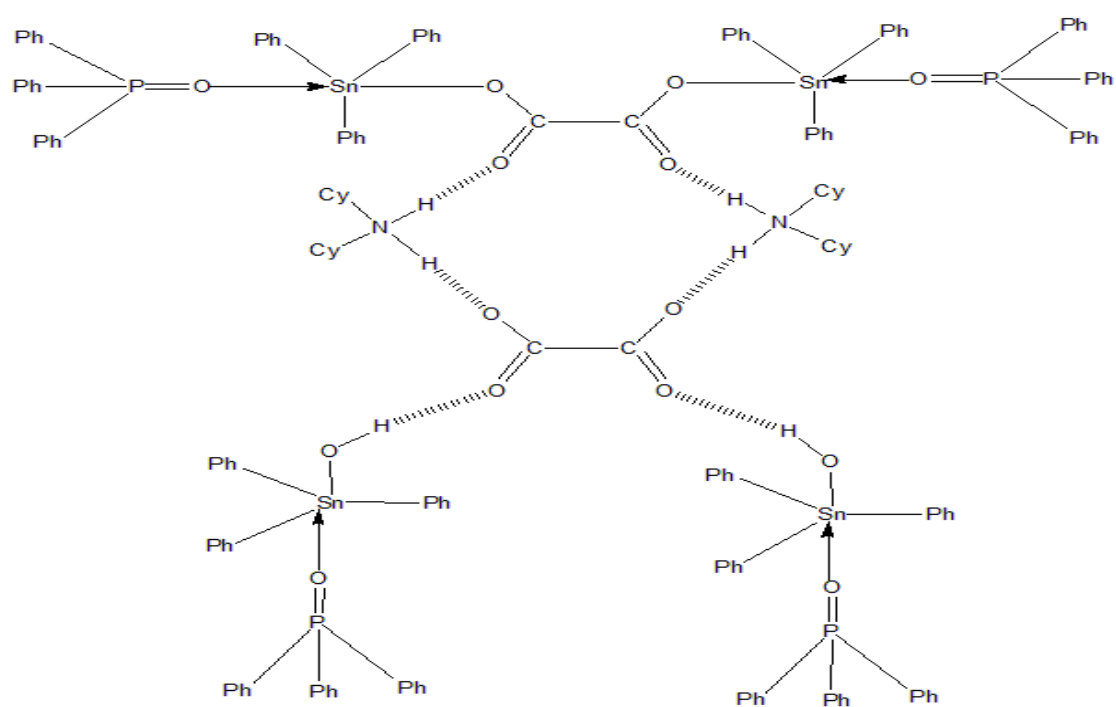


Figure 7a. Proposed structure for compound *G*

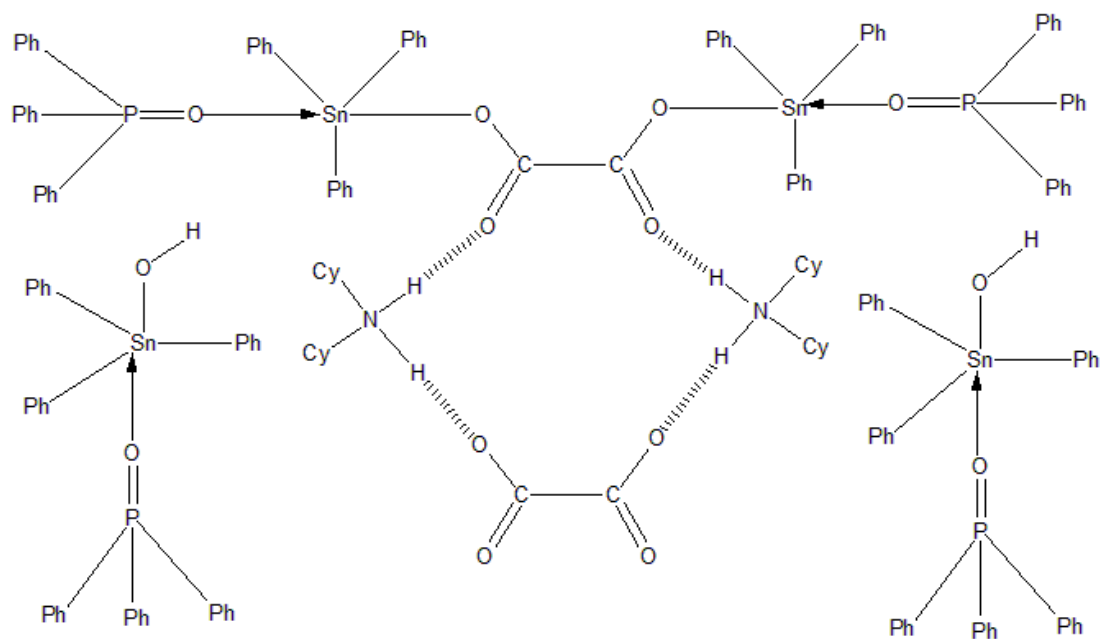


Figure 7b. Proposed structure for *G*

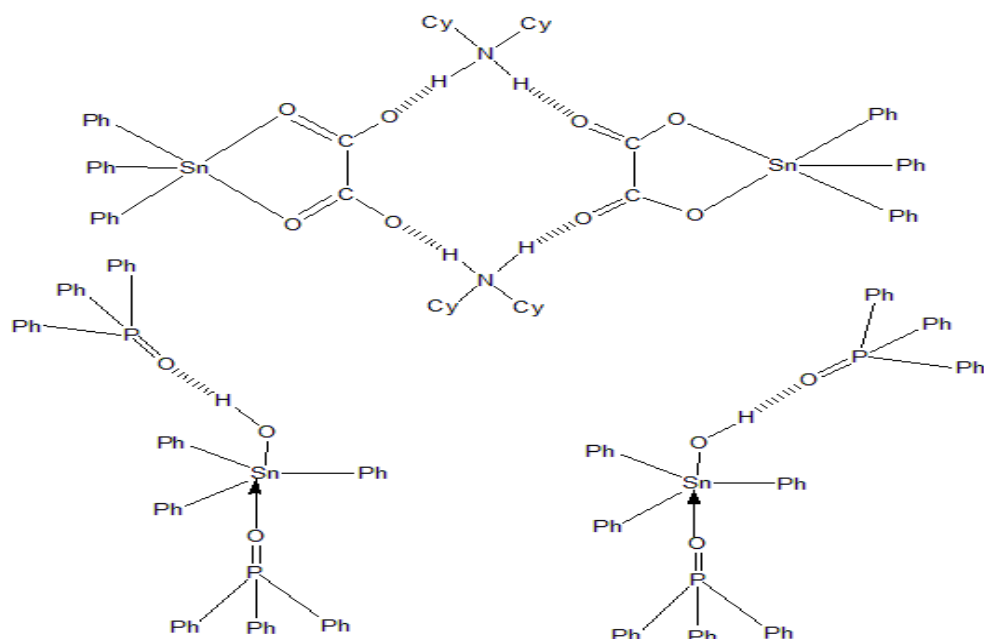


Figure 7c. Proposed structure for G

CONCLUSION

While studied compounds with OPPh₃ contains C₂O₄(SnPh₃)₂ with SnPh₃ residues monocoordinated to a the Lewis base, all the other adducts contain C₂O₄(SnPh₃)₂ with *cis* coordinated SnPh₃ residue characterized for the first time in this work. The oxalate behaves as a mono- or bidentate, a mono- or a bichelating, an only hydrogen bonds involved ligand. In compounds containing none a symmetrical cation, the key role of the cation is noteworthy.

ACKNOWLEDGEMENTS

We thank Professor Dr Raymundo Cea OLIVARES (Instituto de Química-UNAM-Mexico) and Professor M. Vidali - Padova University, Italy - for recording the infrared spectra and performing the elemental analyses.

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