Studii şi Cercetări Ştiinţifice
Chimie şi Inginerie Chimică, Biotehnologii, Industrie Alimentară

2015, **16** (2), pp. 123 – 131

Scientific Study & Research
Chemistry & Chemical Engineering, Biotechnology, Food Industry

ISSN 1582-540X

ORIGINAL RESEARCH PAPER

$R_2NH_2C_2O_4SnC_2O_4SnBu_3$, $(R_2NH_2)_4(C_2O_4)_3(SnR'_3)_2$ (R = Cy, *i*-Bu; R' = Me, Bu) AND SOME MIXED ORGANO- AND HALOTIN (IV) OXALATO NEW MOETIES CONTAINING COMPLEXES: SYNTHESIS, INFRARED, MOSSBAUER AND NMR STUDIES

Yaya Sow, Libasse Diop[*]

Université Cheikh Anta Diop, Faculté des Sciences et Techniques, Département de Chimie, Laboratoire de Chimie Minérale et Analytique (LACHIMIA), Dakar, Sénégal

[*]Corresponding author: dlibasse@gmail.com

Received: March, 30, 2015
Accepted: May, 29, 2015

Abstract: On allowing $(Cy_2NH_2)_2C_2O_4\cdot 2H_2O$ to react with $SnBu_3Cl$ or $SnMe_3Cl$, *i*-$Bu_2NH_2HC_2O_4$ with $SnBu_3Cl$, $(Bu_2NH_2)_2C_2O_4$ with $SnMe_3Cl$, (*i*-$Bu_2NH_2)_2C_2O_4$ with $SnPh_2Cl_2$, a mixture of *i*-$Bu_2NH_2)_2C_2O_4$ and $(Me_4N)_2C_2O_4$ with $SnPh_2Cl_2$, $(Pr_2NH_2)_2C_2O_4$ with $SnPh_3Cl$, the oxalato derivatives and complexes $Cy_2NH_2C_2O_4SnBu_3\cdot 3H_2O\cdot 1/4Cy_2NH_2Cl$, *i*-$Bu_2NH_2C_2O_4SnBu_3\cdot 1/2($*i*-$Bu_2NH_2)_2C_2O_4$,$y_2NH_2C_2O_4SnMe_3\cdot 1/2(Cy_2NH_2)_2C_2O_4$, $Sn(C_2O_4)_2\cdot[C_2O_4(SnMe_3\cdot H_2O)_2]\cdot 2EtOH\cdot 1/4[(Bu_2NH_2)_2C_2O_4]$,(*i*-$Bu_2NH_2)_2\ C_2O_4\cdot 2SnPh_2Cl_2\cdot SnCl_4\cdot 4($*i*-$Bu_2NH_2OH)\cdot 2($*i*-$Bu_2NH_2Cl)$, $2[SnPh_2Cl_4($*i*-$Bu_2NH_2)_2]\cdot SnCl_2C_2O_4\cdot 4$*i*-$Bu_2NH_2Cl$ and $2[Pr_2NH_2C_2O_4SnPh_3]\cdot(Pr_2NH_2)_2C_2O_4\cdot SnCl_4\cdot 2Pr_2NH_2Cl$ were obtained. Double chain, two metallic components and a dimeric structures are suggested with *trans* coordinated $SnMe_3$ residues, these chains being connected through $R_2NH_2^+$ (R = Cy, *i*-Bu) only or by $(R_2NH_2)_2C_2O_4$ bridges. The oxalate behaves as a monochelating, a bridging, an only hydrogen bonding involved or monodentate and H bond involved ligand. The environments around the tin (IV) centres are *cis* or *trans* trigonal bipyramidal, tetrahedral or octahedral. Supramolecular architectures may be obtained when extra hydrogen bonds are possible.

Keywords: *cis or trans trigonal bipyramidal, double chain structure, mono- or bichelating, mono- or bidentate, only hydrogen bonding involved oxalates, tetrahedral or octahedral environments,* *supramolecular architectures, trans coordinated SnR'_3 residues (R'= Me, Bu)*

INTRODUCTION

Many research groups involving our are focusing in attempting to synthesize new molecules belonging to organo- and halotin(IV) family because of several applications found (medecine, industry, agriculture) and also for diversity of structural chemistry [1 - 10]. In this research field we have initiated here the study of the interactions between $i\text{-}Bu_2NH_2HC_2O_4$ and $SnBu_3Cl$ or $SnPh_2Cl_2$, $(Cy_2NH_2)_2C_2O_4\cdot 2H_2O$ and $SnMe_3Cl$ or $SnBu_3Cl$, $(i\text{-}Bu_2NH_2)_2C_2O_4$ and $SnPh_2Cl_2$ $Pr_2NH_2HC_2O_4$ and $SnPh_3Cl$, $(Bu_2NH_2)_2C_2O_4$ and $SnMe_3Cl$ which have given the seven studied derivatives and complexes infrared and NMR studies of which have been carried out, then structures suggested on the basis of the spectroscopic data.

MATERIALS AND METHODS

$(Cy_2NH_2)_2C_2O_4\cdot 2H_2O$ (**L_1**), $i\text{-}Bu_2NH_2HC_2O_4$ (**L_2**), $(Bu_2NH_2)_2C_2O_4$ (**L_3**), $(i\text{-}Bu_2NH_2)_2C_2O_4$ (**L_4**) and $Pr_2NH_2HC_2O_4$ (**L_5**) have been obtained as white precipitates or powders: a solution is obtained mixing aqueous solutions of Cy_2NH, $i\text{-}Bu_2NH$, Bu_2NH or Pr_2NH with $H_2C_2O_4\cdot 2H_2O$ in 2/1, 1/1, 2/1, 2/1or 1/1 ratio respectively. The analytical data of **L_1** to **L_5** are presented in Table 1.

Table 1. *Results of the elemental analyses for L_1 to L_5*

Compound	**Chemical formula**	**Elemental analysis (%)**					
		C		**H**		**N**	
		calc.	found	calc.	found	calc.	found
L_1	$(Cy_2NH_2)_2C_2O_4\cdot 2H_2O$	64.58	65.00	11.61	11.49	5.38	5.26
L_2	$i\text{-}Bu_2NH_2HC_2O_4$	54.77	55.00	9.55	9.49	6.39	6.45
L_3	$(Bu_2NH_2)_2C_2O_4$	62.03	62.00	11.57	11.49	8.04	8.06
L_4	$(i\text{-}Bu_2NH_2)_2C_2O_4$	62.03	62.00	11.57	11.49	8.04	8.06
L_5	$Pr_2NH_2HC_2O_4$	50.25	50.02	8.96	9.01	7.32	7.28

$Cy_2NH_2C_2O_4SnBu_3\cdot 1/4Cy_2NH_2Cl\cdot 3H_2O$ (**A**), $i\text{-}Bu_2NH_2C_2O_4SnBu_3\cdot 1/2(i\text{-}Bu_2NH_2)_2C_2O_4$ (**B**) and $Cy_2NH_2C_2O_4SnMe_3\cdot 1/2(Cy_2NH_2)_2C_2O_4$ (**C**) have been obtained by allowing $(Cy_2NH_2)_2C_2O_4\cdot 2H_2O$ to react with $SnBu_3Cl$ in 1/2 ratio, $i\text{-}Bu_2NH_2HC_2O_4$ with $SnBu_3Cl$ in 1/2 ratio, $(Cy_2NH_2)_2C_2O_4\cdot 2H_2O$ with $SnMe_3Cl$ in 1/1 ratio; a powder was obtained in all cases after a slow solvent evaporation.

$Sn(C_2O_4)_2\cdot[C_2O_4(SnMe_3\cdot H_2O)_2]\cdot 2EtOH\cdot 1/4[(Bu_2NH_2)_2C_2O_4]$ (**D**) and $(i\text{-}Bu_2NH_2)_2C_2O_4\cdot 2SnPh_2Cl_2\cdot SnCl_4\cdot 4(i\text{-}Bu_2NH_2OH)\cdot 2(i\text{-}Bu_2NH_2Cl)$ (**E**) have been prepared by allowing $(Bu_2NH_2)_2C_2O_4$ and $i\text{-}Bu_2NH_2HC_2O_4$ to react with $SnMe_3Cl$ or $SnPh_2Cl_2$ in ethanol in the 1/2 or 1/1 ratio respectively: a white precipitate was obtained in both cases and stirred around two hours. $2[SnPh_2Cl_4(i\text{-}Bu_2NH_2)_2]\cdot SnCl_2C_2O_4\cdot 4i\text{-}Bu_2NH_2Cl$ (**F**) and $2[Pr_2NH_2C_2O_4SnPh_3]\cdot(Pr_2NH_2)_2C_2O_4\cdot SnCl_4\cdot 2Pr_2NH_2Cl$ (**G**) have been obtained by allowing $(i\text{-}Bu_2NH_2)_2C_2O_4$ to react with $SnPh_2Cl_2$ and $Pr_2NH_2HC_2O_4$ to react with

$SnPh_3Cl$ in ethanol in 1/2 and 2/1 ratio respectively; a white precipitate was obtained in both cases and stirred around two hours.

The analytical data of **A** to **G** are presented in Table 2.

Table 2. *Results of the elemental analyses for compound* ***A*** *to* ***G***

Compound	**Chemical formula**	**Elemental analysis [%]**					
		C		**H**		**N**	
		calc.	found	calc.	found	calc.	found
A	$Cy_2NH_2C_2O_4SnBu_3\cdot1/4Cy_2NH_2Cl\cdot3H_2O$	52.78	52.99	9.88	9.76	2.54	2.51
B	$i\text{-}Bu_2NH_2C_2O_4SnBu_3\cdot1/2(i\text{-}Bu_2NH_2)_2C_2O_4$	54.51	54.41	9.96	9.93	4.10	4.15
C	$Cy_2NH_2C_2O_4SnMe_3\cdot1/2(Cy_2NH_2)_2C_2O_4$	54.51	54.49	8.77	8.63	4.24	4.25
D	$Sn(C_2O_4)_2\cdot[C_2O_4(SnMe_3\cdot H_2O)_2]\cdot2EtOH\cdot1/4[(Bu_2NH_2)_2C_2O_4]$	26.59	25.99	4.75	4.86	0.75	0.81
E	$(i\text{-}Bu_2NH_2)_2C_2O_4\cdot2SnPh_2Cl_2\cdot SnCl_4\cdot4(i\text{-}Bu_2NH_2OH)\cdot2(i\text{-}Bu_2NH_2Cl)$	48.67	48.46	8.29	8.24	5.04	5.05
F	$2[SnPh_2Cl_4(i\text{-}Bu_2NH_2)_2]\cdot SnCl_2C_2O_4\cdot4i\text{-}Bu_2NH_2Cl$	47.18	46.91	7.92	7.81	4.89	4.82
G	$2[Pr_2NH_2C_2O_4SnPh_3]\cdot(Pr_2NH_2)_2C_2O_4\cdot SnCl_4\cdot2Pr_2NH_2Cl$	43.44	43.32	5.95	6.03	3.85	3.78

The infrared spectra were recorded at the *Instituto de Química - U.N.A.M., Mexico,* by means of a BX FT-IR type spectrometer. Elemental analyses have been performed at the *Instituto de Química - U.N.A.M., Mexico*.
Infrared data are given in cm^{-1} (IR abbreviations: (vs) very strong, (s) strong, (m) medium, (w) weak, (vw) very weak).
The NMR spectra were recorded in the solid state and in chloroform at the *Instituto de Química - U.N.A.M., Mexico,* by means of a Bruker 300 type spectrometer.
Mössbauer spectra were obtained as described previously [11]. Mossbauer parameters are given in $mm\cdot s^{-1}$ (Mossbauer abbreviations: Q.S = quadrupole splitting, I.S = isomer Shift, Γ = full width at half-height, A = area).
All the chemicals were purchased from Aldrich Company Germany and used as such.

RESULTS AND DISCUSSION

Let us consider the IR and NMR data of the three studied complexes:
$Cy_2NH_2C_2O_4SnBu_3\cdot1/4Cy_2NH_2Cl\cdot3H_2O$

(**A**): $\nu_{as}COO^-$: 1675 (s), 1627 (vs); ν_sCOO^-: 1345 (s),1272 (vs); δCOO^-: 785 (s); $\nu_{as}SnBu_3$: 695 (vs); ν_sSnBu_3: 601 (vw); $i\text{-}Bu_2NH_2C_2O_4SnBu_3\cdot1/2(i\text{-}Bu_2NH_2)_2C_2O_4$;

(**B**): $\nu_{as}COO^-$: 1710 (s), 1650 (vs); ν_sCOO^-: 1345 (s), 1287 (vs); δCOO^-: 779 (s); $\nu_{as}SnBu_3$: 698 (vs); ν_sSnBu_3: 601 (vw); $Cy_2NH_2C_2O_4SnMe_3\cdot1/2(Cy_2NH_2)_2C_2O_4$;

(**C**): $\nu_{as}COO^-$: 1650 (s), 1627 (vs); ν_sCOO^-: 1305 (s), 1282 (vs); δCOO^-: 789 (s); $\nu_{as}SnMe_3$: 555 (vs); ν_sSnMe_3: 518 (vw); NMR Solid-state ^{117}Sn:7.3 ppm; Solution ^{117}Sn: the wide resonance centered in 145 ppm; NMR ^1H ($CDCl_3$): 8.97 (NH); 3.02 (CHCy); 2.07 (CH_2Cy); 1.74 (CH_2Cy); 1.59 (CH_2Cy); 1.20 (CH_2Cy); 0.53 [58/55] (CH_3Sn); NMR ^{13}C ($CDCl_3$): 165.7 (C_2O_4); 53.5 (CHCy); 29.2, 24.9, 24.8 (CH_2Cy); -0.1 (CH_3Sn).

The X ray structures of $Cy_2NH_2C_2O_4SnMe_3$ and $Bu_2NH_2C_2O_4SnMe_3$ have been reported by our group [11]. They consist of chains of $[C_2O_4SnMe_3]^-$ connected as a double chain structure with two bridging cations or as a layered structure with one bridging cation between the chains. The appearance of ν_sSnBu_3 as a weak band is an indication of D3h symmetry allowing to suggest a structure similar to one of those reported by our group in [12] and containing trans coordinated $SnMe_3$ residues (Figure 1).We have arbitrary chosen the double chain structure.

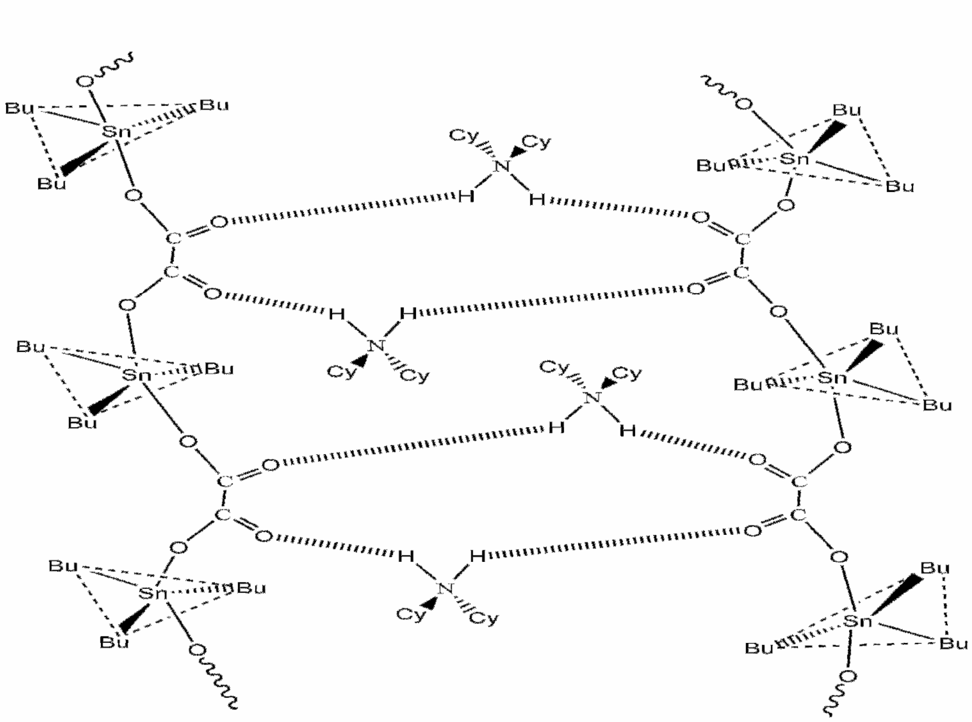

Figure 1.** Proposed structure for compound **A

For (i-$Bu_2NH_2)_2C_2O_4SnBu_3\cdot 1/2(i$-$Bu_2NH_2)_2C_2O_4$ and $(Cy_2NH_2)_2C_2O_4SnMe_3\cdot 1/2$ $(Cy_2NH_2)_2C_2O_4$, the appearance of both ν_sSnBu_3 and ν_sSnMe_3 as weak bands are consistent with trans coordinated $SnBu_3$ and $SnMe_3$ residues allowing to suggest a double chain of $[SnR_3C_2O_4]^-$ connected by anion through $(R_2NH_2)_2C_2O_4$ - R = i-Bu, Cy - *via* NH…….O hydrogen bonds leading to the structure reported on Figure 2.

A second discrete dimeric structure with *trans* coordinated SnR_3 residue can also be considered (Figure 3). The ^{117}Sn value of 7.3 ppm is consistent with the presence of a *trans* coordinated $SnMe_3$ residue - in solution a cleavage of the chains occurs (145ppm).

Let us consider the IR and Mossbauer data:

$Sn(C_2O_4)_2\cdot [C_2O_4(SnMe_3H_2O)_2]\cdot 2EtOH\cdot 1/4[(Bu_2NH_2)_2C_2O_4]$

(**D**): $\nu_{as}COO^-$: 1700 (s), 1670 (s), 1661 (s); ν_sCOO^-: 1280 (vs), 1239 (vs); δCOO^-: 788 (s); $\nu_{as}SnMe_3$, $\nu_{as}SnMe_3$: 520 (vw); $I.S_1$ = 1.27; $Q.S_1$ = 3.71; Γ_1 = 0.9; % A_1 = 67; $I.S_2$ =

0.088; Γ_2=0.94; %A_2 = 33; $(i\text{-}Bu_2NH_2)_2C_2O_4{\cdot}2SnPh_2Cl_2{\cdot}SnCl_4{\cdot}4(i\text{-}Bu_2NH_2OH){\cdot}2(i\text{-}Bu_2NH_2Cl)$;

(**E**): $\nu_{as}COO^-$: 1675 (vs), 1671 (s); ν_sCOO^-: 1299 (vs), 1239 (s); δCOO^-: 790 (s); I.S$_1$ = 0.13; Q.S$_1$ = 0.37; Γ_1 = 0.95; % A_1 = 33; I.S$_2$ = 1.37; Q.S$_2$ = 4.30; Γ_2 = 0.95; % A_2 = 67.

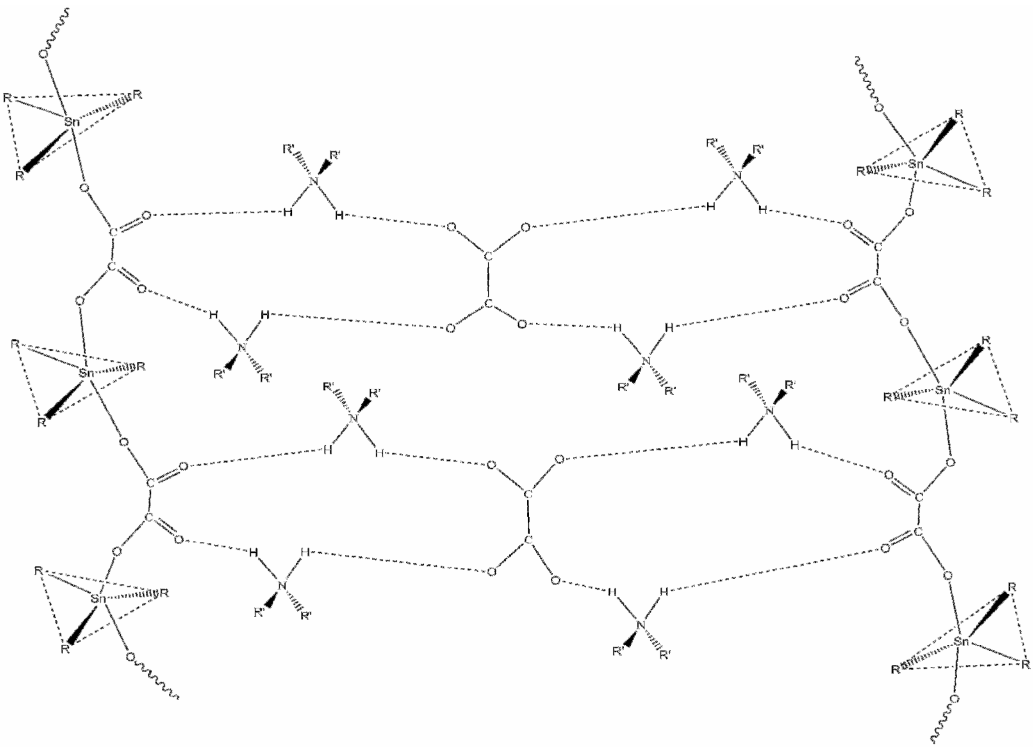

***Figure 2.** Proposed structure for compound **B** and **C** (R = Bu, Me; R' = i-Bu, Cy)*

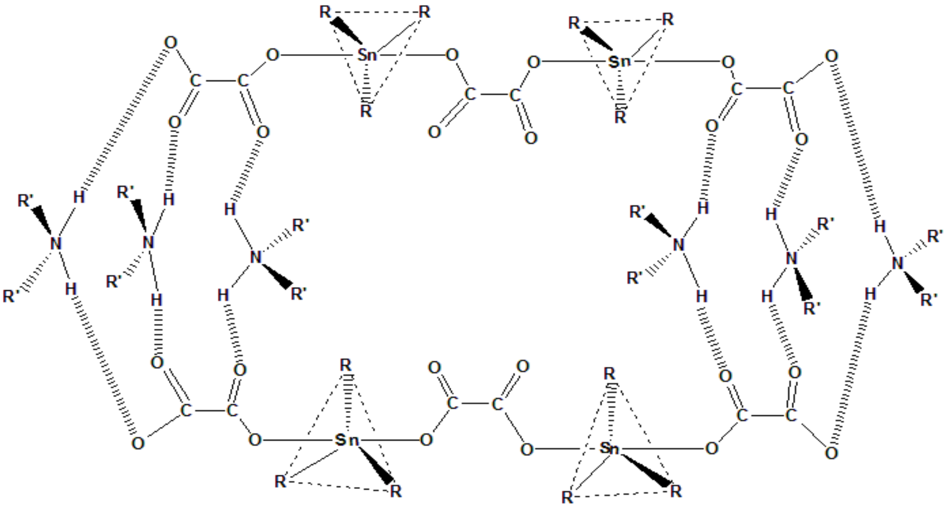

Figure 3.** Dimeric structure suggested for compound **B** and **C

From the Mossbauer data two tin centres are present, one $SnMe_3$ residue in a *trans* coordinated environment, the other one due to the presence of $Sn(C_2O_4)_2{\cdot}2EtOH$ adduct because of the weakness of the isomer shift: a unique absorption as the oxotin(IV) centres and a doublet due to organotin centres allowing to suggest the presence of $C_2O_4(SnMe_3{\cdot}H_2O)_2$ yet reported by the Dakar group [13]. This compound can be considered as a 1/1 adduct between $Sn(C_2O_4)_2{\cdot}2EtOH$ and $C_2O_4(SnMe_3{\cdot}H_2O)_2$. The proposed structure is a two metallic components one: $Sn(C_2O_4)_2{\cdot}2EtOH$ and $C_2O_4(SnMe_3{\cdot}H_2O)_2$ interacting through hydrogen bonds or *van der Waals* forces (Figure 4). A supramolecular architecture may be obtained when extra hydrogen bonds are considered.

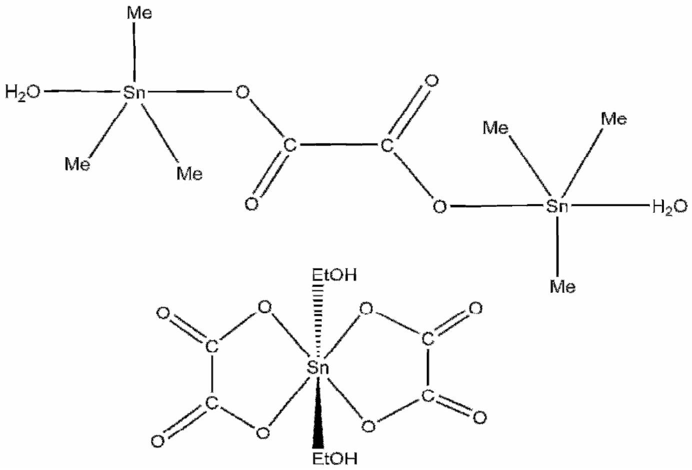

Figure 4. *Proposed structure for* ***D***

The Mossbauer data of this adduct are consistent with the presence of two organotin(IV) centres, one organotin(IV) centre in a *trans* octahedral environment and one halotin(IV) centre according to Bancroft and Platt [14] allowing to conclude to the presence of $[SnPh_2Cl_2(OH)_2]^{2-}$ and $[C_2O_4(SnCl_4)]^{2-}$. The value of the isomer shift of the halotin(IV)centre allows to consider the presence of a SnO_2Cl_4 framework.

The suggested structure is reported on Figure 5. Two Cl^- interact electrostatically with the trimetallic hydrogen bonded component. A supramolecular architecture may be obtained when extra hydrogen bonds are considered.

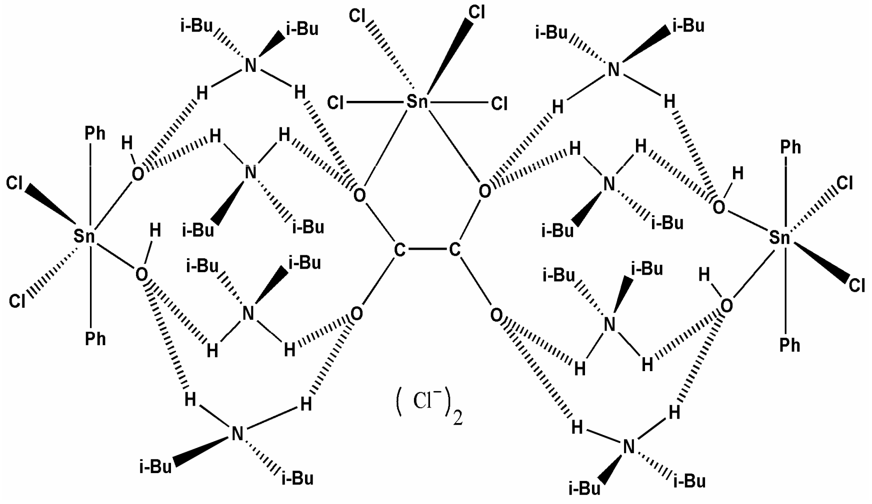

Figure 5. *Proposed structure for* ***E***

Let us consider the IR and Mossbauer data of the studied adducts:
$2[SnPh_2Cl_4(i\text{-}Bu_2NH_2)_2]\cdot SnCl_2C_2O_4\cdot 4i\text{-}Bu_2NH_2Cl$ (**F**): $\nu_{as}COO^-$: 1680 (s), 1652 (vs); ν_sCOO^-: 1251 (vs); δCOO^-: 788 (s); $I.S_1 = 0.61$; $Q.S_1 = 1.92$; $\Gamma_1 = 0.99$; % $A_1 = 66$; $I.S_2 = 0.09$; $\Gamma_2 = 0.97$; % $A_2 = 34$;
$2[Pr_2NH_2C_2O_4SnPh_3]\cdot(Pr_2NH_2)_2C_2O_4\cdot SnCl_4\cdot 2Pr_2NH_2Cl$ (**G**): $\nu_{as}COO^-$: 1647 (vs), 1612 (s); ν_sCOO^-: 1270 (s), 1261 (vs); δCOO^-: 789 (s).
The Mossbauer data of $2[SnPh_2Cl_4(i\text{-}Bu_2NH_2)_2]\cdot SnCl_2C_2O_4\cdot 4(i\text{-}Bu_2NH_2Cl)$ are consistent with the presence of two types of tin centres - two organotin(IV) and one halotin(IV) - the very weak value of the isomer shift of the halotin(IV) centres has not allowed to consider the presence of a O_2SnCl_4 framework according to Tudela [15] equation - $\delta = 0.29 \pm 0.08 mm\cdot s^{-1}$ for chloro compounds - what explain the correlation with the presence of the O_2SnCl_2 framework in the structure. The suggested structure consists of two $[SnPh_2Cl_4]^{2-}$ and $SnCl_2C_2O_4$ components linked by cation …..Cl……..cation hydrogen bonded strings the environments around the tin centres being *cis* octahedral ($1.92\ mm\cdot s^{-1}$) and tetrahedral ($\delta = 0.09\ mm\cdot s^{-1}$) according to Bancroft and Platt [11] allowing to suggest the structure reported on Figure 6.

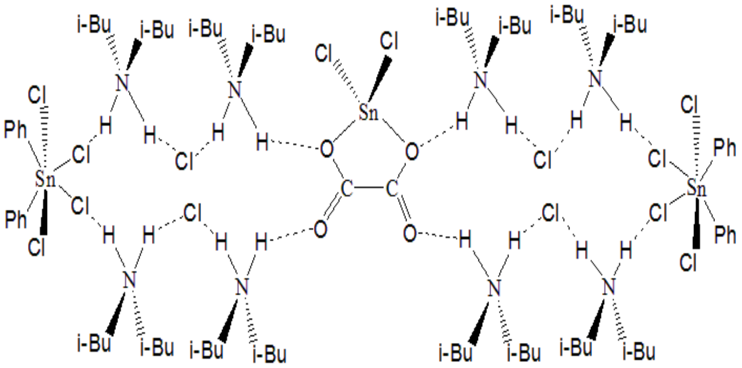

Figure 6. *Proposed structure for compound* ***F***

The structure contains the dimeric $[C_2O_4(SnPh_3)Pr_2NH_2]_2$ - similar to the dimeric structure of $[C_2O_4(SnPh_3)Cy_2NH_2]_2$ reported by Ng and Hook [16] and the complex-anion $[C_2O_4(SnCl_4)]^{2-}$ reported by Sow *et al* [17], these two components being connected by cation……Cl…cation hydrogen bonded strings leading to the structure reported on Figure 7.

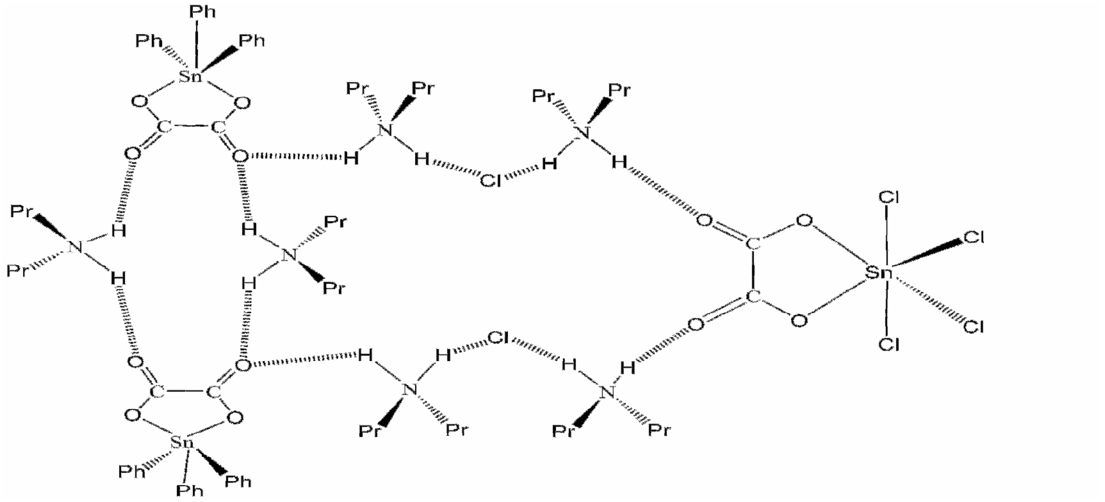

Figure 7. *Proposed structure for compound* ***G***

CONCLUSION

The structure suggested for the studied derivatives are of double chain type connected by cations or $(R_2NH_2)_2C_2O_4$ - R = *i*-Bu, Cy - the SnR_3 residue being trans coordinated, the oxalate behaving as a monochelating, a bidentate bridging ligand or only hydrogen bonds involved. Dimeric or two metallic components structures were also proposed. Supramolecular architectures may be obtained when extra hydrogen bonds are possible.

ACKNOWLEDGEMENTS

We thank Professor José D. ARDISSON from *Laboratorio de Fisica Aplicada*, CDTN/CNEN, Belo Horizonte M.G. Brazil for running the Mossbauer spectra and Dr. Raymundo Cea OLIVARES from *Instituto de Química - U.N.A.M.*, Mexico for recording the infrared spectra and performing part of the elemental analyses.

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